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WRIGHT AIR DEVELOPMENT CENTER

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WADC TECHNICAL REPORT 52-106

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Air Research and Development Command
United States Air Force
Wright-Patterson Air Force Base, Ohio

FOREWORD

This report was prepared jointly by the Metallurgical Research & Development Co., Inc., Washington, D. C., and the S-F-C Research Associates, Patterson, New Jersey, under Contract No. AF 33(038)-16032. The contract was initiated under Expenditure Order No. 460-36-13 SR3A, which was converted to Research and Development Order No. 463-7, "High Temperature Materials Research." It was administered under the direction of the Flight Research Laboratory, Directorate of Research, Wright Air Development Center, with Mr. Murray A. Schwartz acting as project engineer. This report summarizes progress made on this contract during the period 1 October 1950 through 31 March 1952.

ABSTRACT

This summary report covering the period from 1 October 1950 through 31 March 1952 includes the historical background of the flash sintering process, a description of the equipment, and test results. The most favorable conditions of power input, time and pressure required to produce a nickel-titanium carbide compact of optimum physical characteristics was determined. Evaluation of sintered compacts was made by determination of density, hardness, modulus of rupture and optical analysis. Recommendations for further work on this process are made.

PUBLICATION REVIEW

This report has been reviewed and is approved.

FOR THE COMMANDING GENERAL:

LESLIE B. WILLIAMS
Colonel, USAF
Chief, Flight Research Laboratory
Directorate of Research

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SECTION I

INTRODUCTION AND HISTORY

A. Rationalization of Program

Turbine blades are perhaps the most critical component of the gas turbine engine. The thermal efficiency of this engine increases rapidly as parts are developed to withstand higher temperatures. Conventional engines provide for blade operating temperatures in the order of 1500°F maximum, although occasionally the temperature may rise to about 1800°F for a few seconds duration. Manufacturers now provide blades to meet such contingencies, but means are not available to examine blades non-destructively and to establish, prior to application, that they will perform within close predictable limits.

Such materials as appear to satisfy the drastic requirements imposed at temperatures over 1800°F do not lend themselves to easy fabrication by the conventional means of casting, forging, or machining. Methods of hot pressing powder metals, previously investigated, are limited by the inability of die materials to withstand the temperatures and pressures jointly involved. Powder materials offer some advantages in certain areas of the high temperature field, but cannot be considered of much value in the temperature range above 1600°F. Therefore, a method of electric resistance sintering (called flash sintering, by reason of the extremely short time required) was considered as a possible means of solving the problem.

B. Flash Sintering, Defined

Flash sintering, or electric resistance sintering, is defined as the method wherein current conducting powder or preforms pressed from powder are sintered in a die by the application, in a short time interval or intervals, of energy supplied by pressure and current, so regulated and controlled that a predetermined microstructure is attained after sintering.

C. History of Flash Sintering

The process was conceived by Cdr. E. G. Touceda, U.S.N.R., while on duty with the Bureau of Ships during World War II. At the time, it had become apparent that conventional methods of sintering metal powder preforms, such as turbine blades, to close dimension and high density failed to achieve the requirements desired. It was hoped, as a result of preliminary experiments, that the process would provide means to:

1. Obtain extremely high sintering temperatures.
2. Sinter metal powders or metal powder preforms to close dimension, to uniform metallurgical structure, and to high density.
3. Produce blades automatically for gas turbines to close predictable limits of performance.

4. Investigate the sintering characteristics of metal powders having high melting points and to develop new and useful alloys or sintered combinations of metal powders thereby.

Preliminary tests conducted in the Welding Laboratory at Rensselaer Polytechnic Institute prior to 1 July 1946 proved to be so promising that Contract NObs-31493 was awarded to the Institute by the Bureau of Ships. Principal objectives of the contract were to study metallurgical aspects of the process and to investigate means for regulating and controlling the electrical and pressure systems incorporated in the mechanism to achieve desired metallurgical structures in the instance of a wide number of specimens of different powder compositions.

During the life of Contract NObs-31493, from 1 July 1946 to 30 November 1950, a number of metal powder combinations were investigated. Binary powder mixtures such as nickel-chromium and cobalt-chromium were sintered under such conditions as to provide for all stages of element diffusion up to complete melting and extrusion from the die. Both elemental and pre-alloyed powders of the composition of alloy 422-19 were also investigated, as were mixtures of elemental powders with carbides such as silicon carbide or tungsten carbide. Also, single powders such as molybdenum and titanium were sintered to high density and ductility. Later many mixtures of metal powder and non-metallics were investigated as the process was explored as a potential method of producing the so called "ceramets". Objectives were essentially explorative and little attention was directed to the investigation of electrically insulating refractory materials obviously necessary if the process were to be made automatic. Very little attention was directed to the investigation of methods for producing parts of non-uniform section. Considerable attention, however, was given to the development of means of control and instrumentation.

Project Order Nos. 681/47 and 51726/48 were authorized at the U. S. Naval Engineering Experiment Station in Annapolis, Maryland, by the Bureau of Ships to explore the adaptability of the process as a means of producing parts automatically, and to investigate insulating refractory materials. Under this contract, a machine was designed and built, and small cylindrical (1/2" diameter) preforms of 80% nickel - 20% chromium alloy were sintered and ejected automatically. Very little progress was realized in the development of electrically insulating refractories, however.

U. S. Naval Academy Contract N161s19851 and Bureau of Ships Contract NObs-50110 were awarded to the Lukens Steel Company to explore the adaptability of the process as a means of producing large (3" diameter) forgeable ingots from metal powder preforms. Ingots of molybdenum, titanium, 422-19 alloy, and binary powder mixtures were sintered and forged. However, further investigations are necessary to obtain uniform control of microstructure before the method can be considered commercially acceptable for the production of these larger sizes.

Lack of available funds has terminated the projects at the Engineering Experiment Station and Lukens Steel Company, but the work at Rensselaer Polytechnic Institute has been extended through Contract NObs-55219 to explore the adaptability of the process as a means for producing sintered carbide tool materials.

Certain limited investigations, conducted under Contract NObs-31493, indicated the Flash Sintering Process might have merit as a method of producing ceramets. This would be especially true if means could be found to improve the electrical conducting properties of metal powder and ceramic powder mixtures having resistance too high to allow for the passage of current at low voltages, in the order of from 4-40 volts, when compressed under loads of the order of 10,000-20,000 psi. Consequently, a proposal to investigate the foregoing was submitted to the Air Materiel Command in June 1950. This was accepted substantially as presented and work commenced on 1 October 1950, although at the request of the Air Force in June 1951, the emphasis of the program was directed to sintering cemented titanium carbide compositions to the exclusion of certain other objectives.

SECTION II

SINTERING MECHANISM

A. Operational Objectives

Sintering of metallic powders by rapid heating through direct passage of electric current of high amperage with simultaneous application of compressive force was initially attempted on conventional spot welding equipment. The results were not too satisfactory as in general, resistances of powders are of a much higher order than those present in sheet metal and the compression of a powder compact during the sintering process is much greater than is experienced in the compression of two sheets of metal being spot welded. Therefore, the flash sintering of powders required the design of an electric flash sintering machine differing in a number of ways from the conventional spot welder although it is unquestionably closely related to it in many respects.

Since spot welding equipment has been used extensively and its full description may be found in a number of publications, such as the Welding Handbook published by the American Welding Society, a general familiarity with its construction and method of operation is assumed. It should be sufficient merely to point out that a spot welder is essentially a press, the platens of which are connected to a low voltage-high amperage transformer, thus permitting a compressive force and an electrical voltage to be applied simultaneously to any substance placed within its jaws. While still employing the basic elements of the spot welder, the following objectives were considered to be of greatest importance in designing the present flash sintering equipment:

1. Maximum flexibility in duration and magnitude of both compressive force and applied voltage.

2. Minimum friction and inertia of movable parts to assure maximum acceleration and maintenance of pressure (follow-up), even in case of rapid consolidation of the compact being sintered.

3. Ability to vary the voltage to be applied to the compact being sintered essentially instantaneously (from the first pulse to the second) in order to compensate for rapid variation within the compact or between compacts of different composition.

The desired objectives were presented to Sciaky Brothers, Inc., Chicago, Illinois, manufacturers of welding equipment, and a joint design was evolved which has satisfied reasonably well the needs of this investigation to date. The sintering machine (Figure 1) consists essentially of a 10 ton air-actuated four-post press with one movable and one stationary copper platen connected to a 440/40-20 volt transformer controlled by means of an electronic switch.

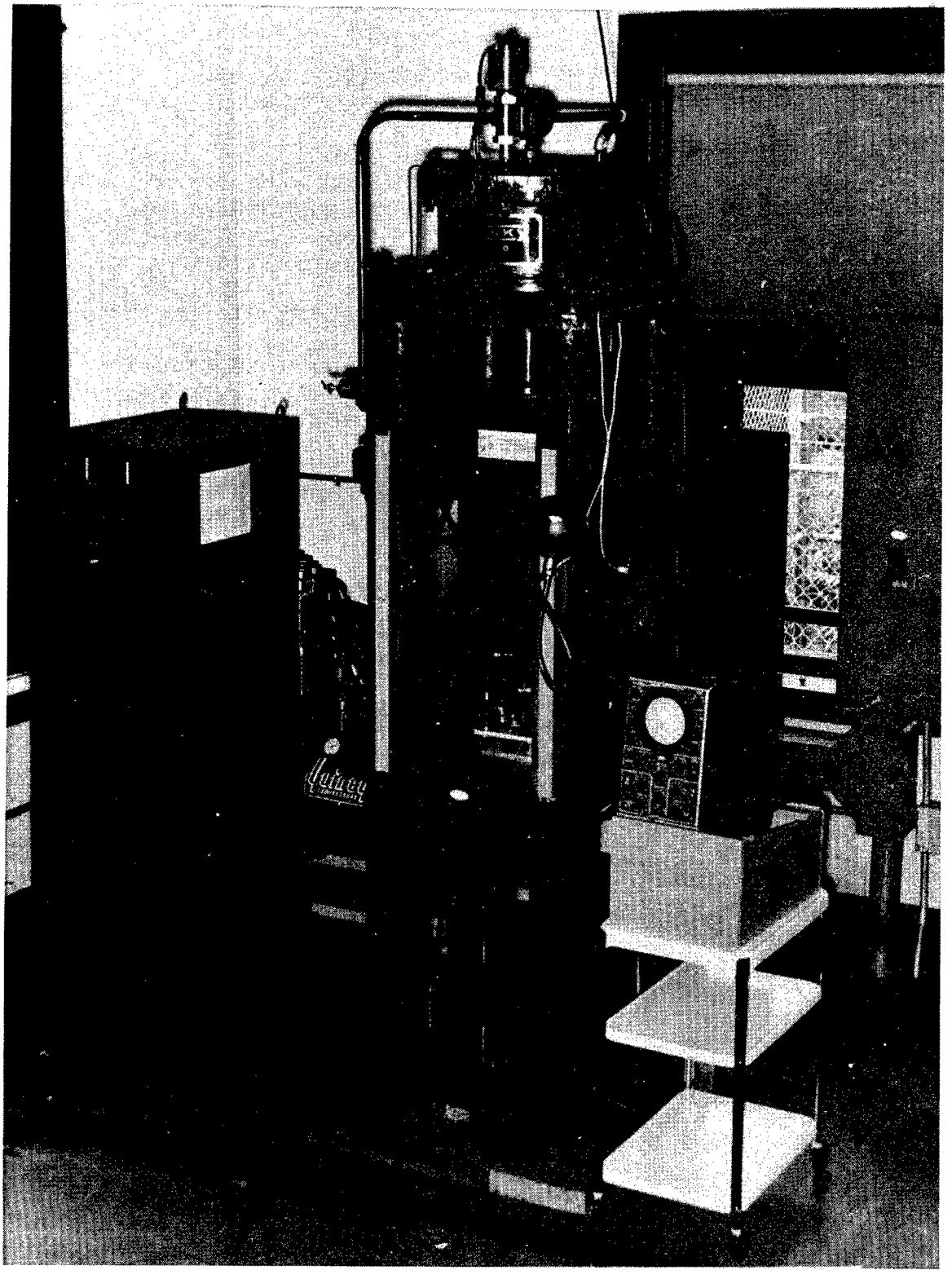


Figure 1. Flash Sintering Machine and Auxiliary Equipment

1. The movable platen of the press is guided on roller bearings and a compressible rubber pad 1/2" thick is inserted just back of the copper platen. This pad, when compressed, provides a source of extremely rapid-acting elastic energy. The press is operated by a double stroke cylinder providing a total movement of 6". It is controlled by three pressure regulators which permit admittance of air at two separate pressures into both top and bottom chambers, with maintenance of virtually constant pressure in the bottom chamber throughout the length of the stroke. Thus, the piston may operate on the differential pressure between the top and bottom chamber which permits very fine control of the compressive force exerted by the press and renders the system independent of the weight of the piston and the movable platen. An additional feature is provided which permits rapid exhaust of the bottom chamber at the end of the stroke or at any desired time, which results in a two stage force; that is, it is possible to apply an initial force of a certain magnitude and then, at any desired point in the sintering cycle, to superimpose upon it a further predetermined force.

2. The electrical system consists of a transformer with a series-parallel winding on both primary and secondary sides connected to a tap changing switch and providing a total of eight voltage steps with approximately equal spacings. The magnetic and electrical capacity of the transformer are such that it will deliver forty volts on open circuit and a total of 20,000 amperes. The electrical impedance at sixty cycles of the secondary circuit with the platens short-circuited is approximately 300 micro-ohms. The welding transformer is supplied from a 4160 volt line through a 150 KVA transformer and an air breaker switch. It is controlled through an ignitron switch and an electronic circuit which permits application of two independently controlled, both in duration and in magnitude, impulses of voltage, separated by an intermediate period, also of controlled length. The magnitude of the secondary voltage is accomplished by a phase shifting device, while the duration of one impulse and of the intermediate period is accomplished by a mechanical relay, and the duration of the second impulse by means of a thyratron circuit. All time periods may be controlled in one cycle (1/60 second) steps up to a total of thirty cycles duration. The electronic control further permits complete application of the pressure cycle without energizing the electrical power circuit and permits continual

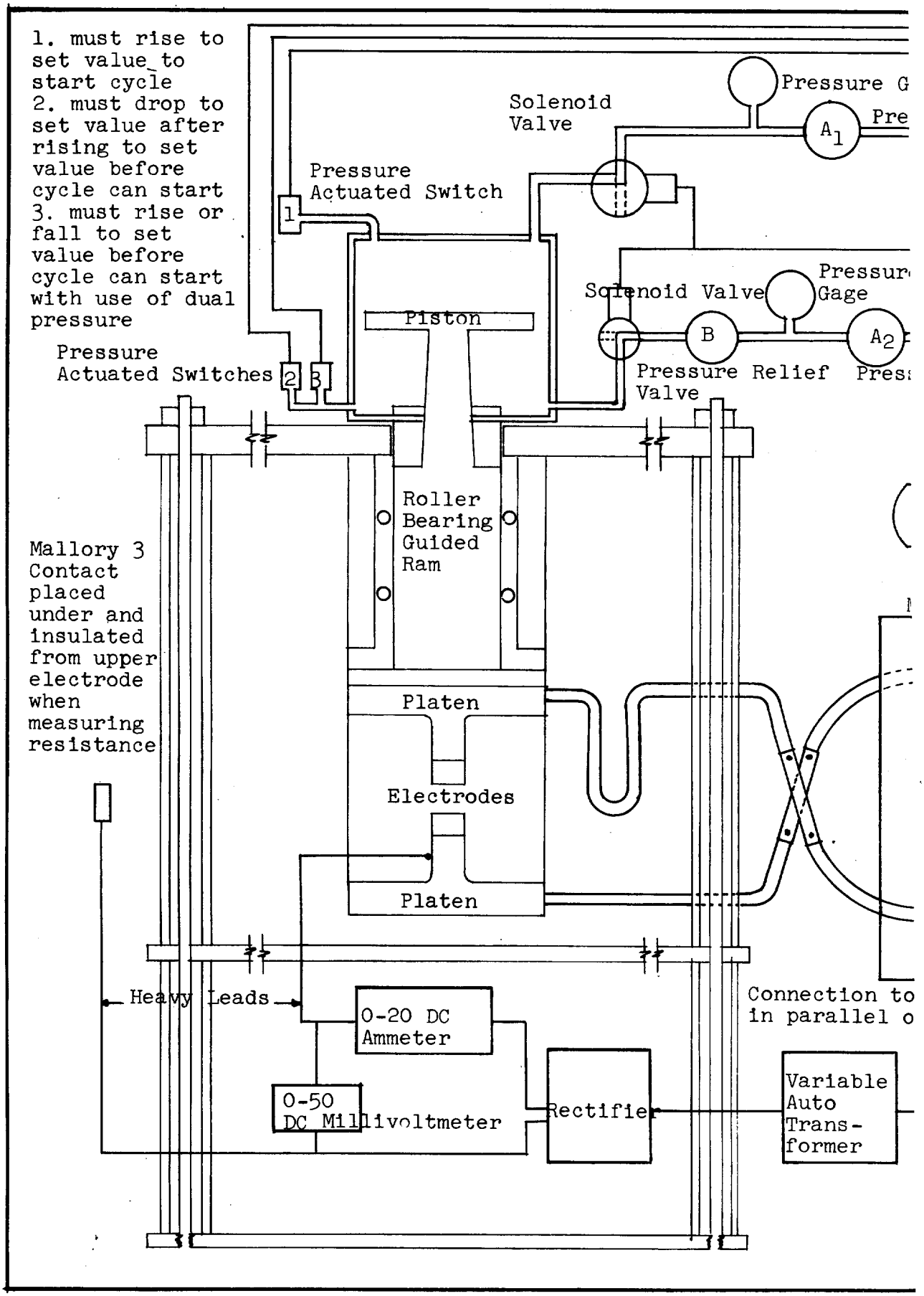
application of pressure after sintering or a release of pressure at a predetermined period after sintering.

A more detailed description of the construction and method of operation of the equipment may be derived from the diagrams and detailed specifications appearing in the succeeding portion of this section.

B. Function of Components of Mechanism

ELEMENTS AND THEIR FUNCTION	REQUIREMENTS OF ELEMENTS	MEANS USED TO MEET REQUIREMENTS	PERFORMANCE CHARACTERISTICS
1. Current Conducting and Pressure Application System			
(a) Pressure Control Valves and Gages			
Provide means for admitting compressed air to actuating piston assembly and control the pressure sufficiently to provide a ram load of from 0-20,000 lbs., and to indicate the pressure.	Air admission and exhaust valves must be quick acting. Pressure control valves must maintain constant pressure during entire length of stroke and pressure conditions must be reproducible from the flash sintering of one specimen to another. Gages must have rapid response and be accurate.	The pneumatic circuit employed for control is shown as part of Fig. 2. Pressure regulators A_1 A_2 are set to provide the desired pressures above and below the piston. The resultant difference in pressure causes the ram to move. A pressure relief valve B prevents build-up of pressure in the lower part of the cylinder above the setting of regulator A_2 piston when the head is lowered prior to initiation of the sintering cycle. Regulation of the speed of stroke is thus controlled. During the sintering operation, solenoid valves operate so as to control the head without restriction on its speed.	The regulator valves are over compensated in the lower part of their range causing a pressure variation of 3 psi in the ram chambers, corresponding to about 750 lbs. ram load. Recently gages, accurate to 1/2 psi, have been installed and it is now possible to reset the regulator valves for each specimen.
(b) Movable Current Conducting Pressure Head			
Provide means for applying pressure and current to contact plungers and maintaining complete alignment during the pressure cycle.	Pressure head must accurately follow the compact as its dimensions change during flash sintering and it must maintain perfect alignment during the complete cycle.	Alignment is provided with roller bearing guides for the ram to which the movable current conducting pressure head is attached through a rubber pad with insulated bolts. The rubber pad serves to store energy and provide for immediate follow-up during the sintering cycle. A water cooled removable electrode of Mallory #3 metal is attached by screw threads to a platen fixed to the movable head of the machine.	The system appears to be in good alignment and the ram moves freely. Electrodes have been replaced when damaged by expelled specimens.
(c) Fixed Conducting Pressure Head			
Provide a rigid support and conduct current to the sintering assembly.	Fixed head must be rigid and free of vibration. Current conducting faces must be of minimum resistance and sufficiently strong to prevent distortion.	A removable water-cooled electrode of Mallory #3 metal is screwed to a platen rigidly attached to the main frame of the machine and insulated from it.	The system appears to be in good alignment. Electrodes have been replaced when damaged by expelled specimens.
(d) Electrical Control			
Provide means of applying voltage to the conducting heads of the flash sintering machine, at predetermined values for predetermined intervals of time, and coordinate electrical and mechanical action.	Control requirements are rigid and reproducibility is of primary importance. Control must be flexible and accurate at all settings.	(1) Main Current Control. The machine transformer is provided with 8 tap settings at equal voltage intervals (see Fig. 2). (2) Control for Time and Heat. This is illustrated diagramatically in Fig. 2. The heat control employs a conventional phase shift circuit using whichever potential divider the relays insert. A synchronous timer is used for single pulses, for the first of two closely spaced pulses, and for the last of two widely spaced pulses, whichever is chosen. Manual switches permit a choice of alternatives. A hold timer keeps the current conducting pressure head closed after current has ceased to flow. Relays can actuate valves to increase the pressure at the end of the first pulse if desired. (3) Recording System. The recording system is also illustrated diagramatically in Fig. 2. It comprises a DuMont 304-H oscilloscope having a long persistance screen and fitted with a DuMont 35 mm camera. Shielded leads from a shunt in the main line bring a signal which triggers the sweep and records the current during the pulse. When the movable pressure head drops past a switch, the trace intensity is changed and this change is recorded in the picture showing when rapid collapse and densification of the compact occurs. (4) Resistance Measuring System. Circuit used for measuring resistance is illustrated as part of Fig. 2. Resistance of the whole sintering assembly is measured before sintering each specimen. One lead is attached to the fixed head and another to a plate insullated from the movable head and placed between it and the upper plunger.	The main current control response of the equipment satisfies design requirements. Timer calibration checked each day shows that the timers faithfully reproduce their settings. Tests made at heat settings of 20, 40, 60, and 80% of full scale show the currents were reproducible within 2% of the value on repeated tests at single settings and within 4% when a repeated setting to the same value was made from upscale by downscale (see Fig. 3). Peak current values are proportional to settings as shown in Curve 1. When the percentage duration of current in the cycle is allowed for, after the methods of Nippes and Savage,1/ it is seen in Curve 2., that the RMS value is not quite linear. With more resistance in the circuit and the resultant smaller current a more linear relationship is obtained. The oscilloscope image appears to be a faithful record of the current variation although subject to slight distortions when current magnitude is increasing rapidly. The image on 35 mm film was enlarged to 2 ft. high for measurement and an internal standard voltage which was constant to about 3% was used for reference (see Fig. 4). Measurements show that a variation of about 5% is obtained in the resistance measuring system employed. Tests of a number of 20% titanium carbide - 80% nickel specimens shown in Fig. 5, illustrate the amount of scatter, certainly due in part to variations of machine load and variations in the processing of the compacts to be sintered.
2. Sintering Assembly			
(a) Current Conducting Plungers and Contact Wafers			
Provide means of transmitting current and pressure heads of the flash sintering machine to the compact within the sintering die and liner and confine the compact material within the die as it undergoes changes in physical or chemical state.	Plungers must have sufficient strength at operating temperature to resist deformation and they must be of high conductivity and thermal capacity are advantageous when cooling at the compact-wafer interface is to be avoided. In operation, wafer resistance and shape control temperature distribution during sintering.	Mallory #100 or Mallory #3 alloy is used for plungers. They are machined to a diameter slightly less than 1/2" and are about 1-1/2" long. Wafers are machined to .500 or .490" in diameter depending upon the requirements to fit a given liner. They are about 5/16" thick and are made of tungsten when sintering the titanium carbide and nickel powder mixtures. In the sintering of nickel and alumina compacts, stainless steel wafers can be used. To prevent sticking the contact face of the wafer is coated with graphite "Dixonac".	Mallory #3 operates well as plungers with loads up to 8000 lbs. Over 8000 Mallory #100 must be used. As plunger ends tend to soften, they must be remachined occasionally. Plungers must move freely in the liner to avoid jamming. At loads up to 12,000 lbs. tungsten wafers function well. In excess of 12,000 lbs. however they tend to crack and deform. Since the sintered material rises between the wafer and the liner, it must be cleaned from the wafer after each use.
(b) Sintering Die and Liner			
Provide means for allowing current conducting and pressure transmitting plungers to exert their energy in a confined area on the compact to be flash sintered and to do so without contamination of the compact; also provide means of flash sintering a compact to predetermined dimensions and to a desired micro-structure.	Dimensional stability of the die and its liner is of primary importance as is the chemical and refractory inertness of the contact surface of the liner with respect to the particular materials being flash sintered. When liners are used, available materials must be chosen which are capable of being molded or machined to close dimensions. Liners should have low thermal and electrical conductivity and high resistance to abrasion.	Clamping dies machined from two brass blocks are used to grip the ceramic liners of AlSiMag 35. Some tests have been made using AlSiMag 202 liners and in some instances metal dies have been used with various dielectric surface coatings. In spite of the fact that liners are specified to be 1/2" I.D. and 3/4" O.D. they are many times elliptical and shims must be used to provide a proper fit.	Ceramic liners vary as much as .007" in internal diameter so that sorting is necessary in order to provide liners into which wafers and compacts will fit without jamming. AlSiMag 202 has been discarded as it is too porous and absorbs metal from the compact during sintering.
(c) Sintering Assembly Support			
Provide means of holding die assembly in a suspended position until the pressure heads close upon the current conducting plungers, permit double action pressure effect by allowing downward movement of die assembly, and support the assembly upon release of the pressure heads.	Support must be aligned with the pressure heads and electrodes to allow the electrodes to distribute their pressure equally on the current conducting plungers.	The sintering assembly support consists of a bakelite plate or table having four corner bearings which slide on 3/4" rods (Fig. 6). The weight of the assembly is balanced by springs. Spacer blocks are used to raise the die assembly above table level, if necessary, to allow space for the plungers.	The support moves freely. Die alignment is checked visually.

1/E. Nippes and W. Savage - "Instrumentation for Flash Welding" - Proceedings of Second Conference on Resistance Welding - American Institute of Electrical Engineers - 1950


1. must rise to set value to start cycle
2. must drop to set value after rising to set value before cycle can start
3. must rise or fall to set value before cycle can start with use of dual pressure
Pressure G
Pre
Solenoid Valve
A1
Pressure Actuated Switch
1
Solenoid Valve
Pressur
Gage
Piston
B
A2
Pressure Actuated Switches
2
3
Pressure Relief Valve
Pres
Roller Bearing Guided Ram
Mallory 3 Contact placed under and insulated from upper electrode when measuring resistance
Platen
Electrodes
Platen
Heavy Leads
0-20 DC Ammeter
Connection to
in parallel o
0-50 DC Millivoltmeter
Rectifier
Variable Auto Trans-former

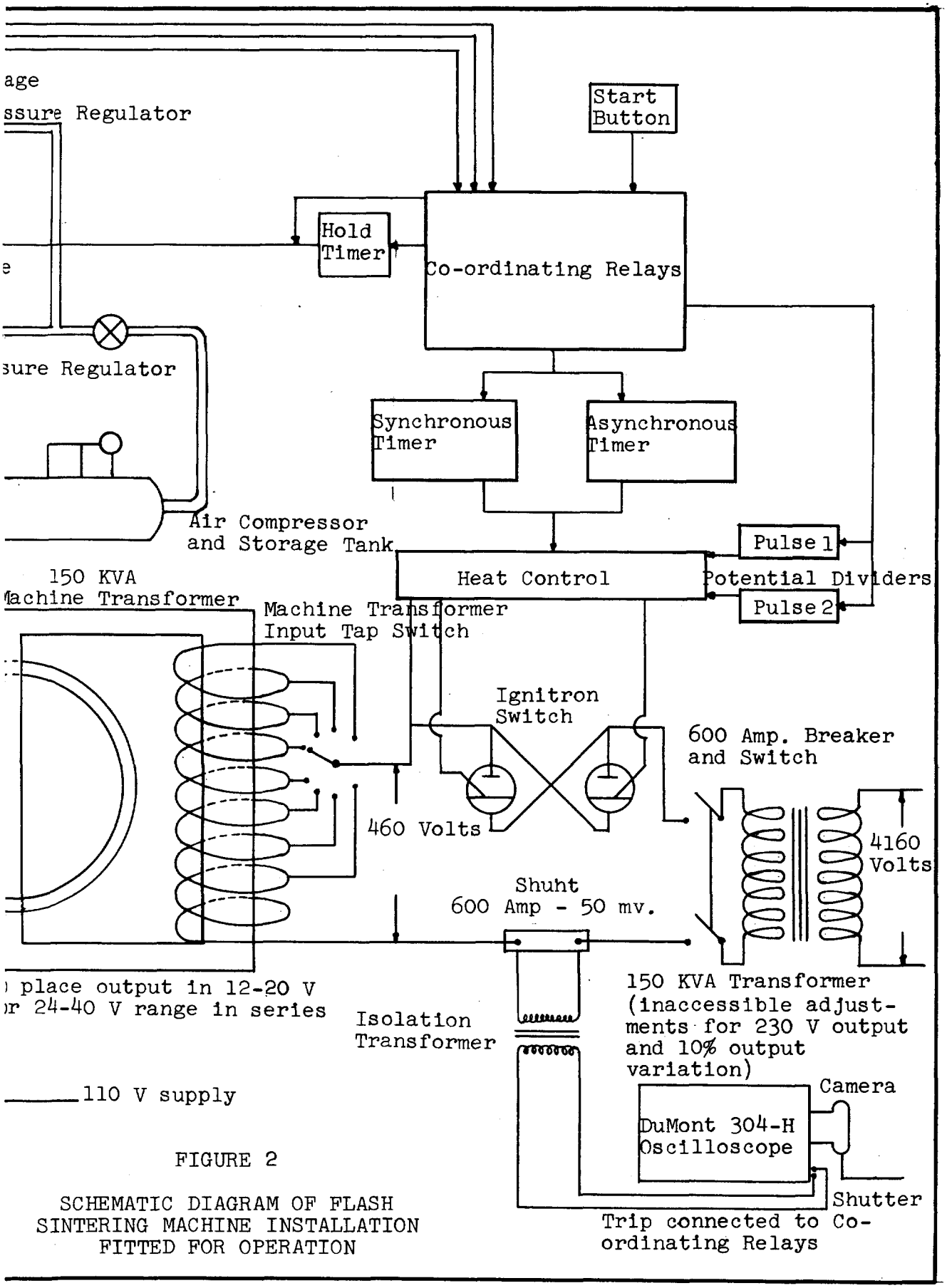


FIGURE 2

SCHEMATIC DIAGRAM OF FLASH SINTERING MACHINE INSTALLATION FITTED FOR OPERATION

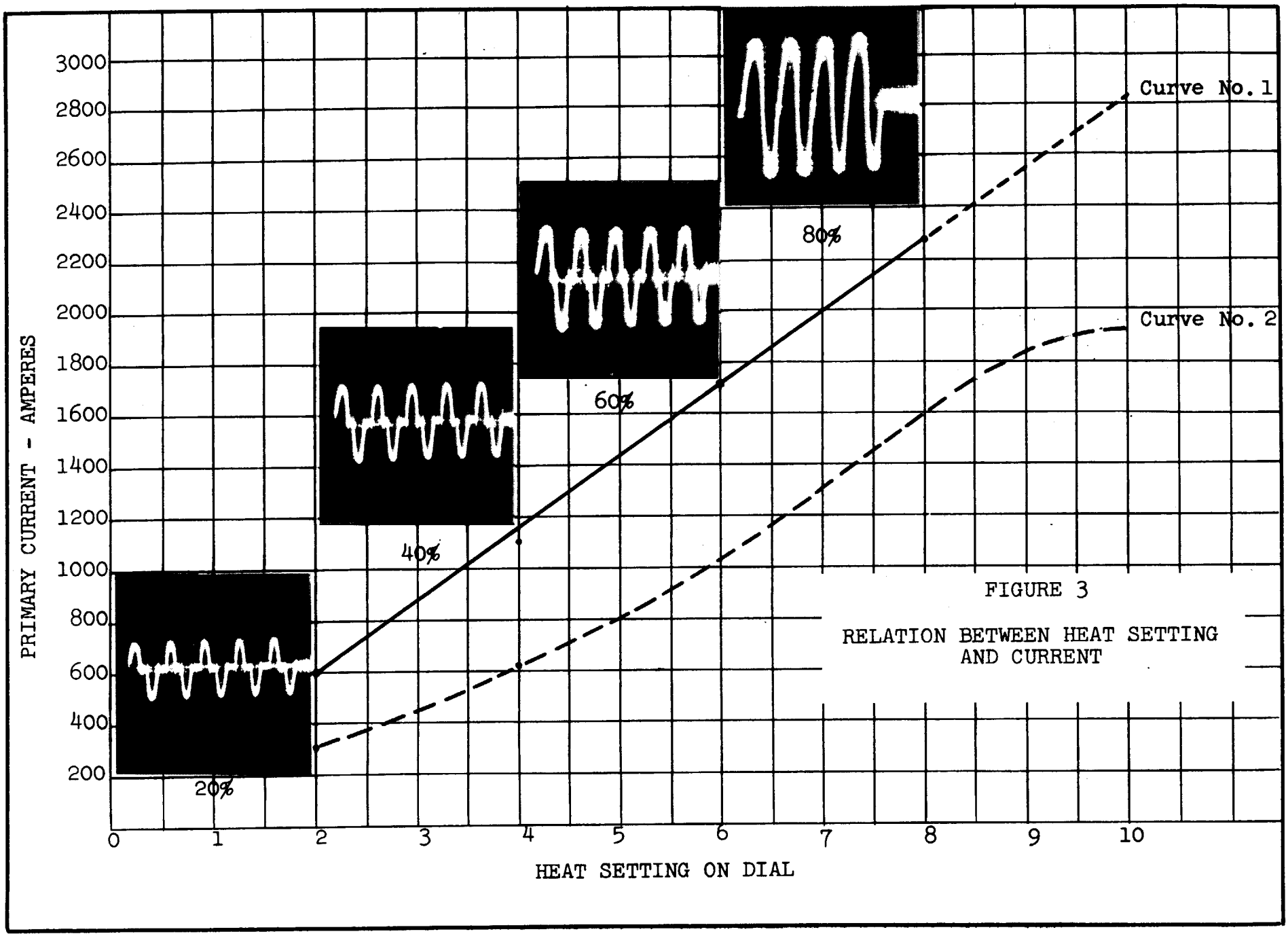


FIGURE 3

RELATION BETWEEN HEAT SETTING AND CURRENT

FIGURE 4

TYPICAL SINTERING PULSES SHOWING THE EFFECT OF VARIOUS SEQUENCES IN OPERATION

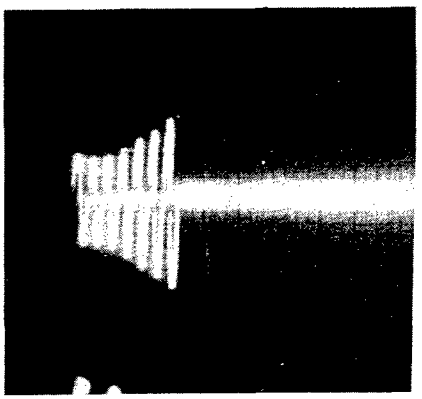


Figure A
Short pulse - 100% heat set. Current rises rapidly after establishing conduction in first two cycles.

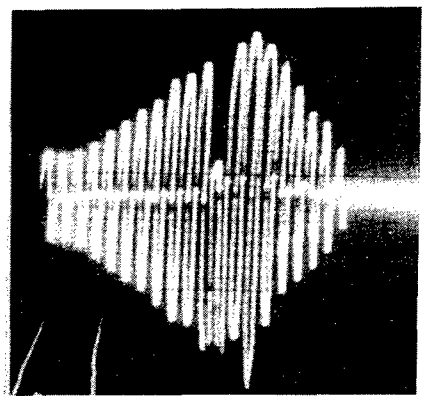


Figure B
Longer pulse - 100% heat set. Increased energy caused melting at about the eighth cycle. Most of the compact had been expelled after the eleventh cycle.

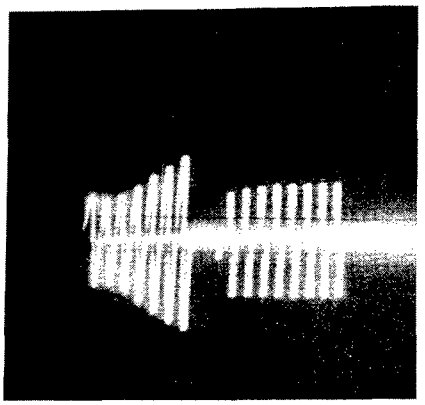


Figure C
Two pulses - two cycle interval between pulses. First pulse 100% heat set, second pulse 20% heat set.

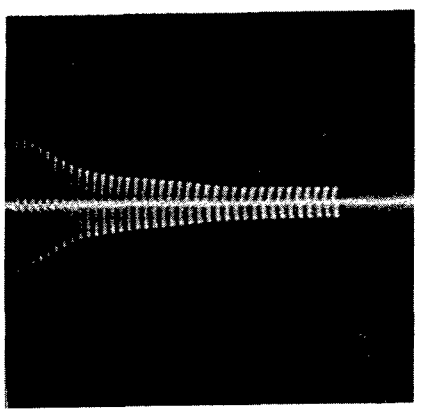


Figure D
Long pulse - 30% heat set recorded with slow sweep.

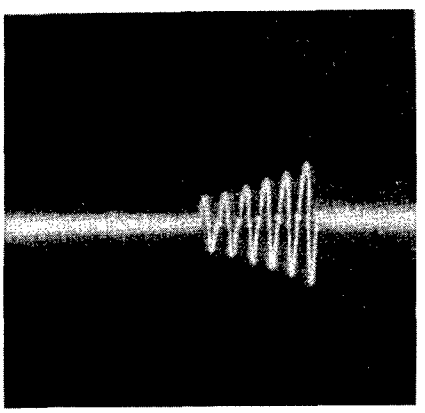


Figure E
Short pulse - 90% heat set. Low sintering pressure of compact has caused high resistance. Current increases in this instance slowly.

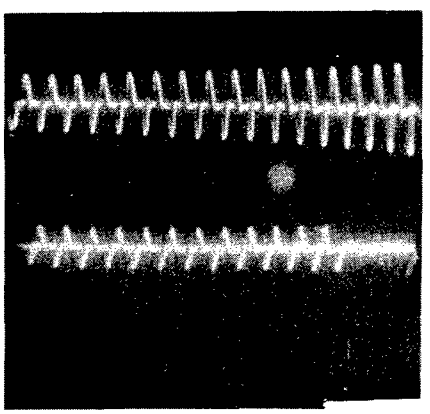


Figure F
Two pulses - 3.5 seconds interval between pulses. First pulse 65% heat set (top graph). Second pulse 35% heat set (bottom graph). With this treatment the temperature of the compact tends to equalize between pulses.

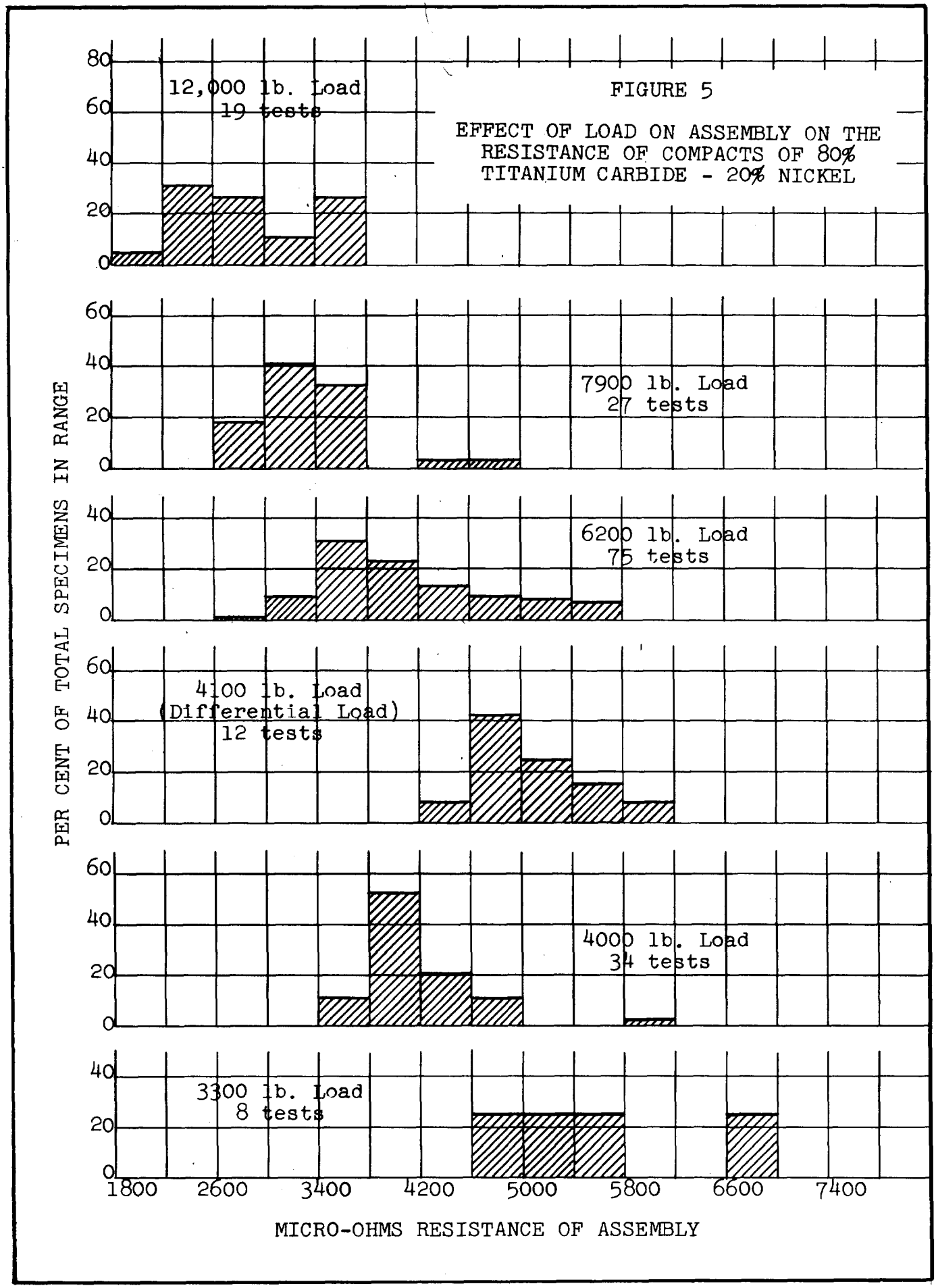
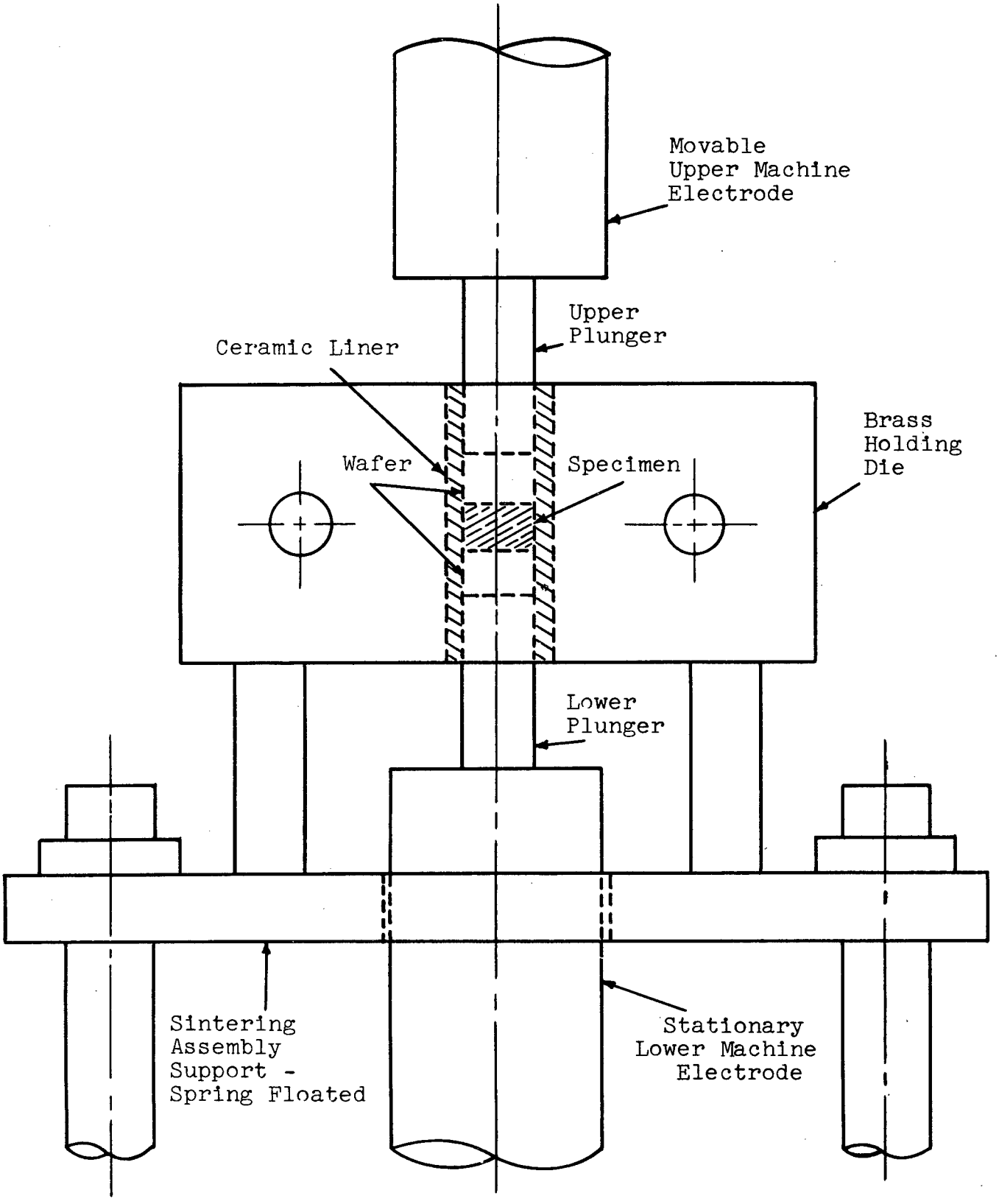


FIGURE 5

EFFECT OF LOAD ON ASSEMBLY ON THE RESISTANCE OF COMPACTS OF 80% TITANIUM CARBIDE - 20% NICKEL

FIGURE 6. ASSEMBLY FOR FLASH SINTERING



SECTION III

COMPACT PREPARATION PRIOR TO FLASH SINTERING

A. Resistance Requirements

In the process, sintering occurs within a sufficiently short time interval so that the metal in the compact is not appreciably oxidized. Maximum voltage at the machine electrodes is held to 40 volts based on present design. Theoretically, therefore, with a current flow of 1000 amperes, compacts of a resistance of 40,000 micro-ohms, could be sintered within these limitations. Means of producing compacts of minimum resistance were investigated prior to the arrival of the sintering machine, as it was anticipated that compacts of lower resistance would sinter more uniformly.

Sufficient current to effect sintering can be passed without difficulty through compacts of pure conductors. However, problems associated with uneven current distribution may arise. Resistance increases as larger percentages of semi or non-conductors are added; however, the increase in resistance is not directly proportional to the amount of non-conductor present, but increases either exponentially or in stepwise fashion. At some point, a small addition of non-conductor produces a great increase in resistance. Since many refractory materials are non-conductors, it is desirable to know how much of these may be present without greatly lowering the conductivity. It is also desirable to know if there are ways of treating compacts so that their resistance can be lowered to permit passage of sintering current and to promote more uniform and rapid flash sintering. Compacts with a specific resistance of as much as 1700 micro-ohm centimeters have been sintered successfully when the total resistance was over 7000 micro-ohms. Test results are shown in Section IV. It is believed that research conducted during the resistance measuring phase of the project will be of definite assistance in planning the direction to be taken in subsequent investigations. For instance, it probably will be necessary to employ different techniques to initiate current flow through different combinations of metals and refractories.

B. Equipment and Procedure for Measuring Resistance

Apparatus used for measuring resistances in conjunction with the flash sintering machine is shown diagrammatically in Figure 2. However, in the earlier work, resistance measurements were taken with this same apparatus using the 10,000 lb. range of a hydraulic testing machine for applying loads to compacts of metal-alumina compositions. Compacts tested at low loads (of the order of 1000 lbs. and less) were placed between two flat contacts of Mallory 3 metal insulated from the testing machine. (At these low loads, support of the compact in a die normally is not needed.) Compacts whose resistance was to be measured at higher loads were confined in the sintering assembly (Figure 6) supported loosely on rubber stoppers on the testing machine. In this instance, electrodes 1/2" in diameter were inserted in the ends of the die for use as the contact plungers between the specimen and the flat contacts of the resistance testing apparatus. The entire assembly was confined in a standard 1/2" I.D. ceramic die liner.

Three identical specimens were tested under the same conditions for each given sequence of compact preparation variables. The compacts tested were 1/2" diameter by 3/8" long and were formed initially from blended powders by double action pressing at 50 tsi. Resistance measurements obtained at various load increments were generally within ± 2-1/2% of the mean value.

C. Experience in Measuring Resistance

Compact Behavior

In the resistance measuring assembly, intimate contact at the specimen-plunger interface is established over the initial portion of the loading range. Reproducibility of results in this range was improved by careful preparation of the contact surfaces of both compact and plungers. As the load was increased to a range of the order of 1000 lbs., no significant changes in the physical structure of the specimen were apparent. However, as the load was increased further, typical 45° shear failure of the specimen was found to occur. When confined in a die, however, following such failure, the specimen would be recompacted at higher loads, still retaining much of the internal structural characteristics of the original specimen. This was established by visual examination of numerous specimens. If at any point in the loading, the load was removed and then re-applied, the load vs. resistance curve was found to have been shifted to higher values of resistance for a given load. Successive reapplications of loads caused the curve to approach limiting values of resistance.

Resistance Change with Increasing Load

Figure 7 shows that in most instances, following an initial drop, resistance of metal-alumina combinations decreases very little with increasing load after application of a preliminary low load. The measurements depicted in Figure 7 and all other resistance determinations (except as noted) are the average of three values which were taken on specimens approximately 0.4" long. Curves for loading in the lower ranges are shown for 80% titanium carbide - 20% nickel in Figure 8. Figure 9 shows the same effect in the high load range on a series of 1/2" diameter 80% titanium carbide - 20% nickel specimens made from single compacts of different lengths.

Effect of Compact Length on Resistance

In Figure 9, it may be seen that the rate at which the resistance changes with load apparently is a function of the length of specimen. Using the same data shown in Figure 9 and plotting resistance vs. length for a series of loads (see Figure 10), a straight line relationship is indicated for lengths up to 1/2". Departures from a straight line relationship above 1/2" are probably due to some differences in density between short and long compacts caused by wall resistance in pressing. (The resistance at zero length is the resistance of the die assembly alone, which also varies somewhat with load.) Appreciable checking has shown that, in general, resistance is proportional to length. It appears likely that a direct proportion would exist for one-half inch diameter specimens with lengths in excess of one-half inch if such specimens consisted of a series of compacts having maximum

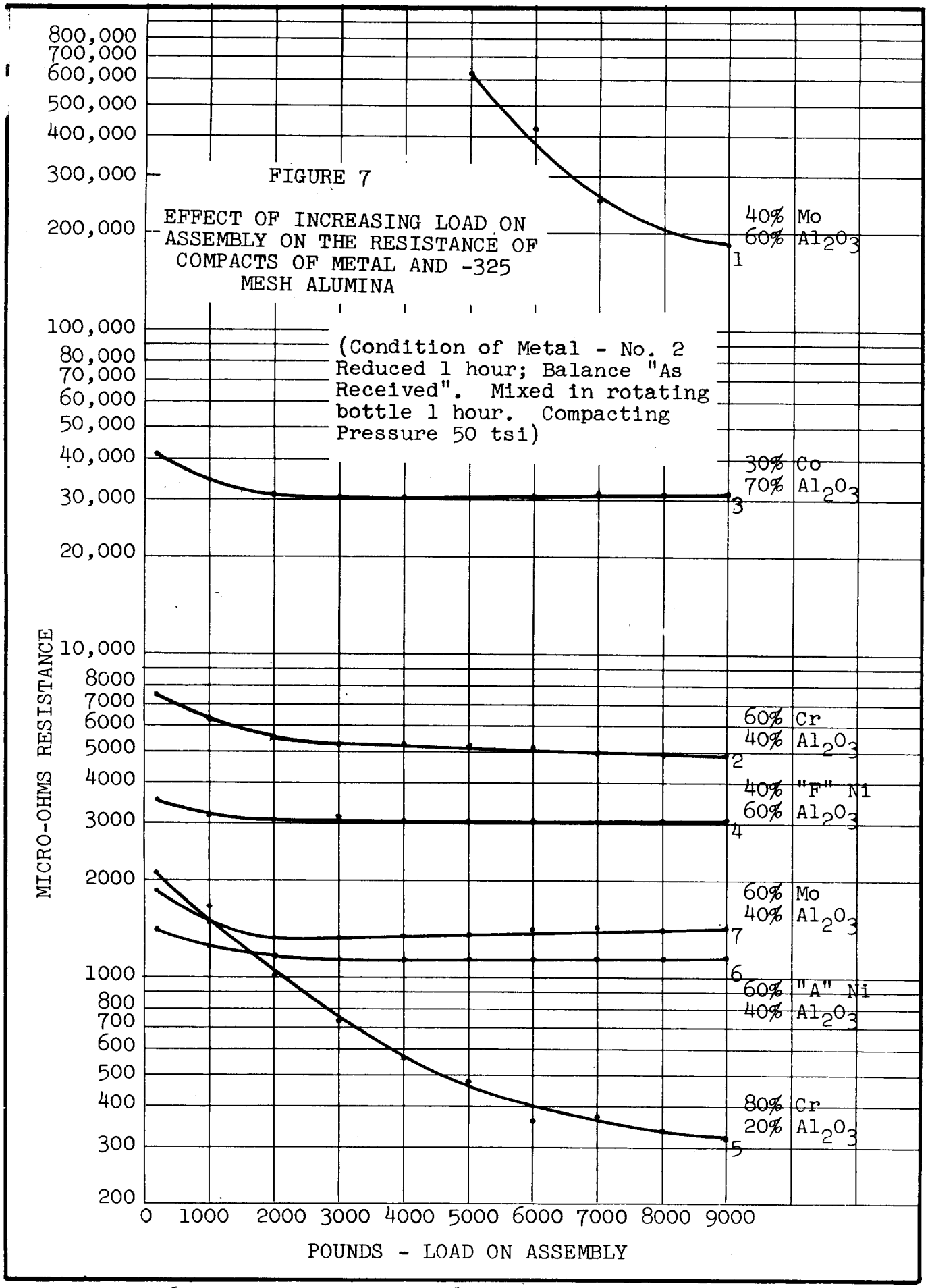
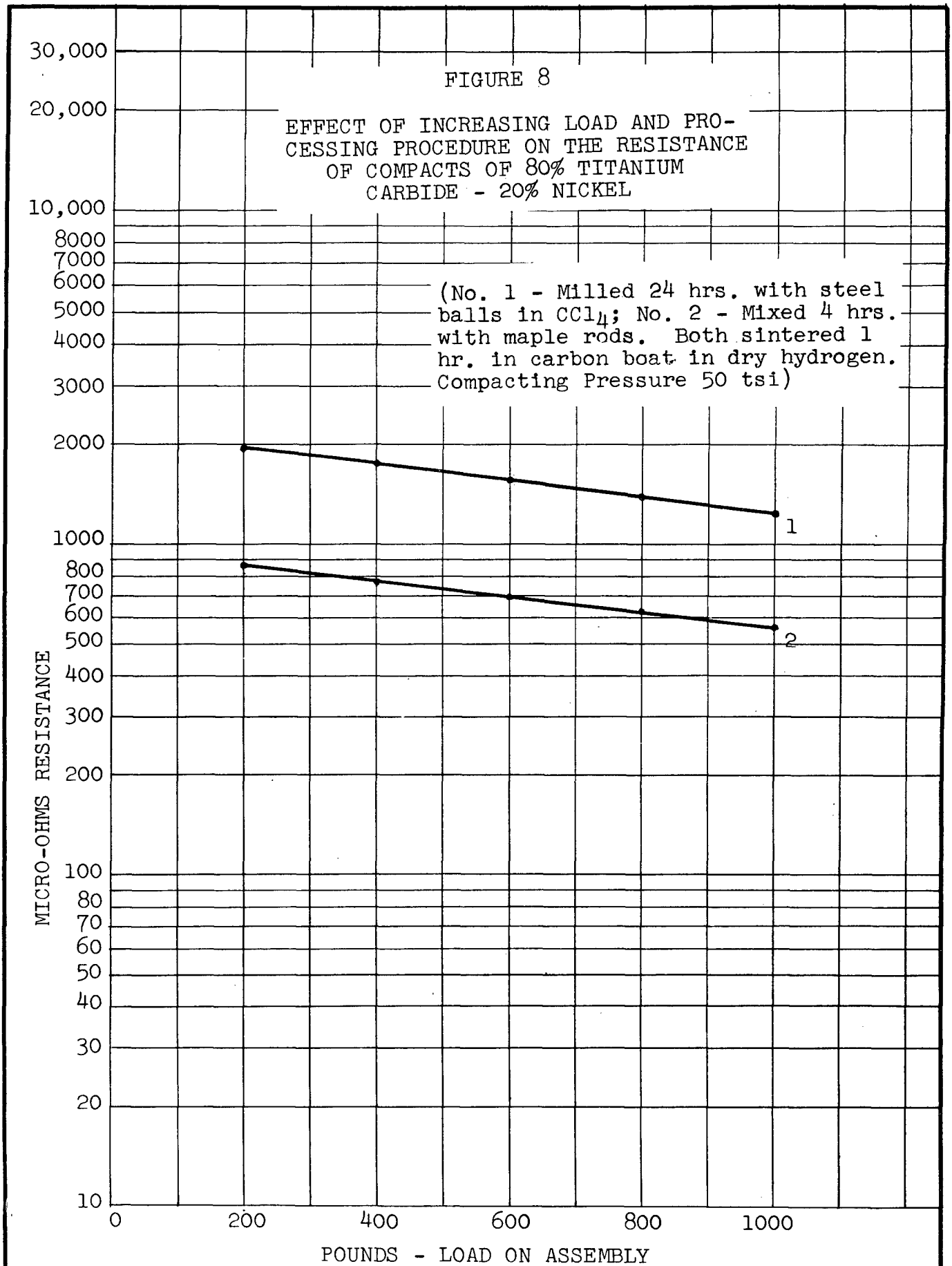


FIGURE 7
EFFECT OF INCREASING LOAD ON ASSEMBLY ON THE RESISTANCE OF COMPACTS OF METAL AND -325 MESH ALUMINA
(Condition of Metal - No. 2 Reduced 1 hour; Balance "As Received". Mixed in rotating bottle 1 hour. Compacting Pressure 50 tsi)
MICRO-OHMS RESISTANCE
800,000
700,000
600,000
500,000
400,000
300,000
200,000
100,000
80,000
70,000
60,000
50,000
40,000
30,000
20,000
10,000
8000
7000
6000
5000
4000
3000
2000
1000
800
700
600
500
400
300
200
0 1000 2000 3000 4000 5000 6000 7000 8000 9000
POUNDS - LOAD ON ASSEMBLY
40% Mo
60% Al2O3
1
30% Co
70% Al2O3
3
60% Cr
40% Al2O3
2
40% "F" Ni
60% Al2O3
4
60% Mo
40% Al2O3
7
6
60% "A" Ni
40% Al2O3
80% Cr
20% Al2O3
5

FIGURE 8

EFFECT OF INCREASING LOAD AND PROCESSING PROCEDURE ON THE RESISTANCE OF COMPACTS OF 80% TITANIUM CARBIDE - 20% NICKEL



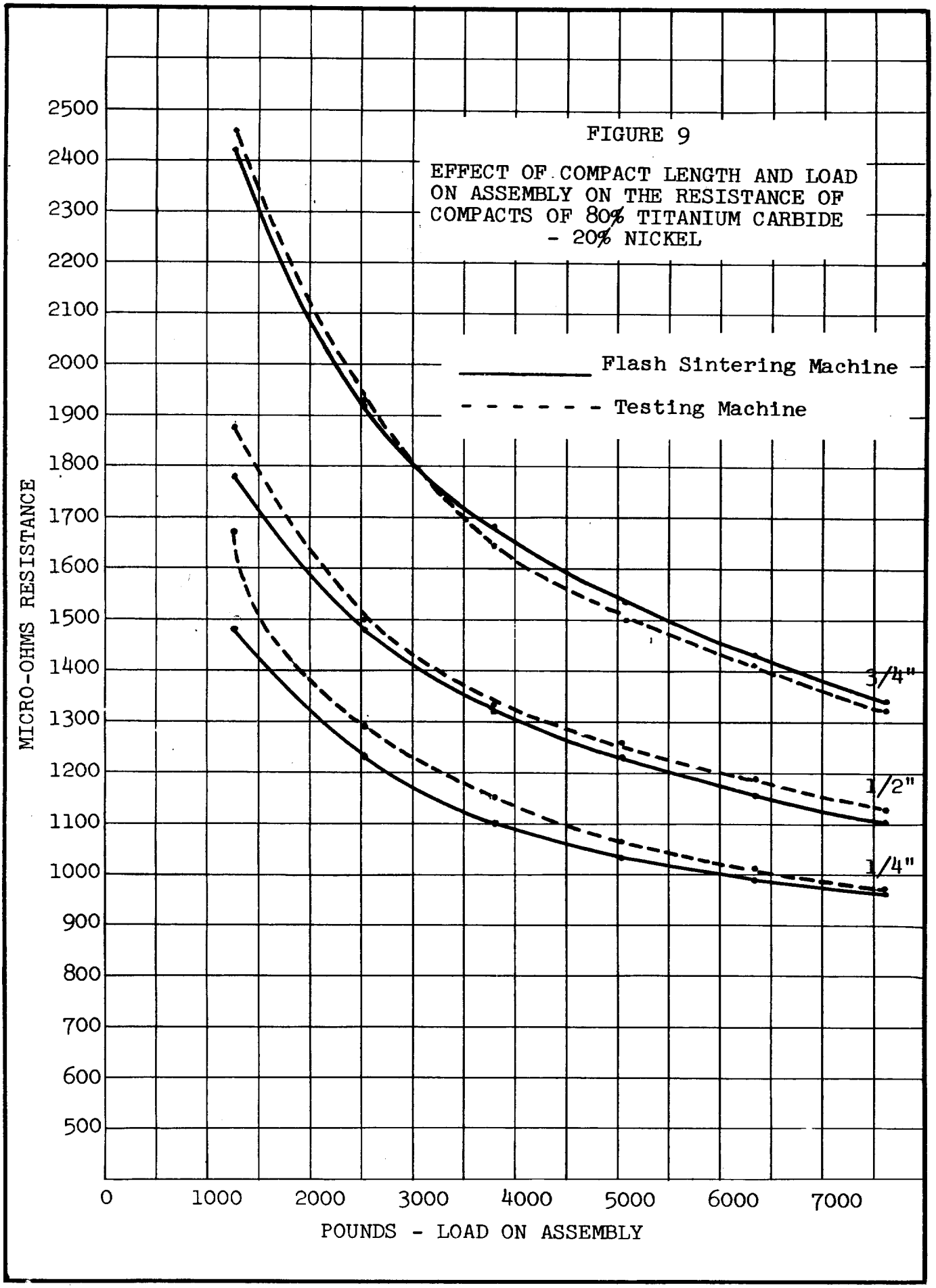


FIGURE 9
EFFECT OF COMPACT LENGTH AND LOAD ON ASSEMBLY ON THE RESISTANCE OF COMPACTS OF 80% TITANIUM CARBIDE - 20% NICKEL
Flash Sintering Machine
Testing Machine
3/4"
1/2"
1/4"
MICRO-OHMS RESISTANCE
2500
2400
2300
2200
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
0
1000
2000
3000
4000
5000
6000
7000
POUNDS - LOAD ON ASSEMBLY

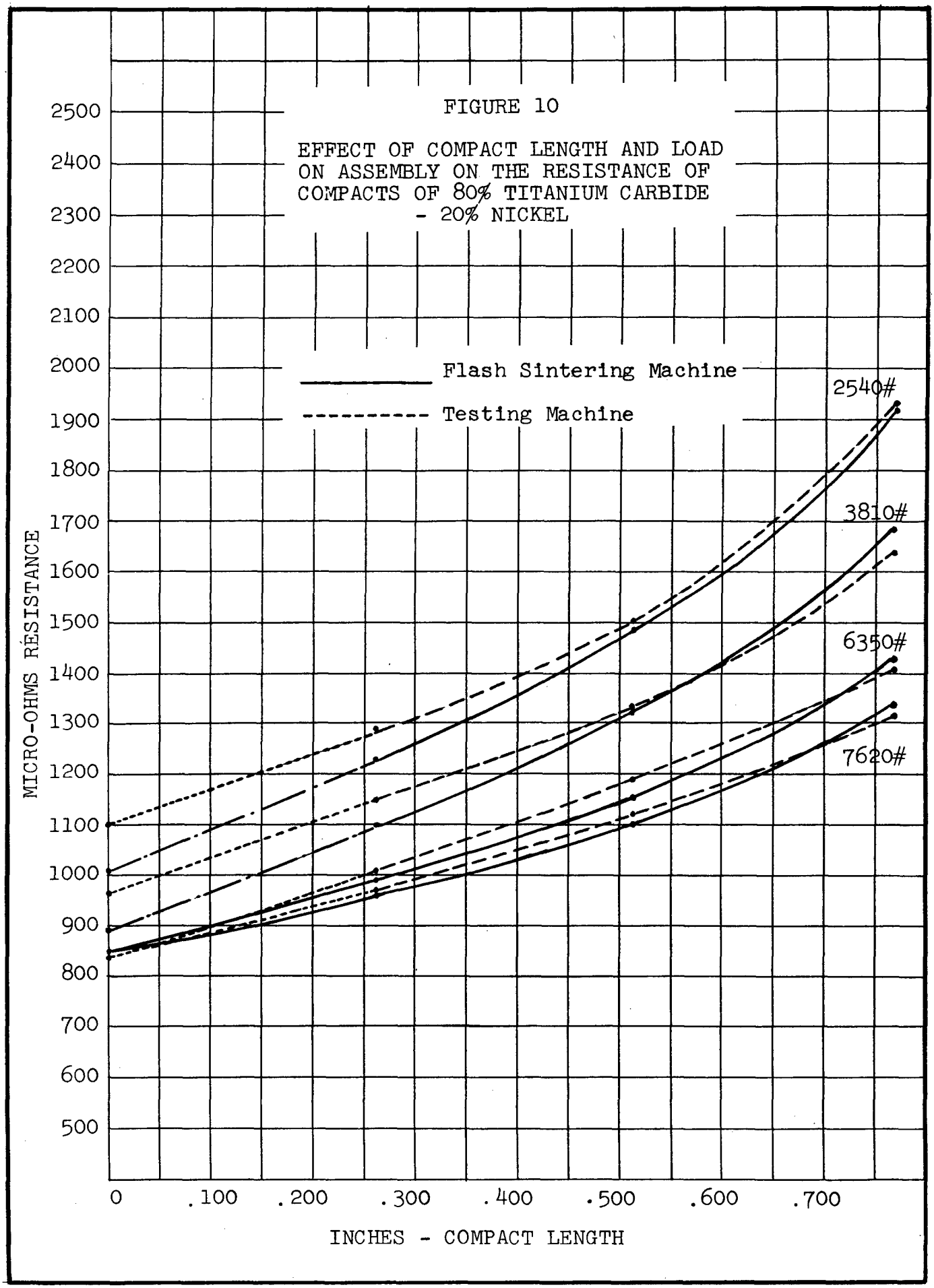


FIGURE 10
EFFECT OF COMPACT LENGTH AND LOAD ON ASSEMBLY ON THE RESISTANCE OF COMPACTS OF 80% TITANIUM CARBIDE - 20% NICKEL
Flash Sintering Machine
Testing Machine
2540#
3810#
6350#
7620#
MICRO-OHMS RESISTANCE
2500
2400
2300
2200
2100
2000
1900
1800
1700
1600
1500
1400
1300
1200
1100
1000
900
800
700
600
500
0
.100
.200
.300
.400
.500
.600
.700
INCHES - COMPACT LENGTH

individual lengths of one-half inch. Measurements with various currents indicate that, so long as the current does not produce heating, the resistance is independent of the current used in the measurement.

D. Metal-Ceramic Systems

Choice of Materials

Investigations were initiated with metal-alumina systems, for the following reasons: (a) reaction and transformations are at a minimum in these systems, (b) alumina powder is conveniently handled, and (c) alumina is a good representative non-conductor as resistances at room temperatures are not likely to differ with different non-conductors having particles of the same size and shape characteristics. Nickel, cobalt, molybdenum, and chromium were chosen as metals since they are readily available in powder form and experience has shown them to be of interest in ceramet compositions. The characteristics of the powders used are given in Table I of the Appendix.

Powder Characteristics

The most noteworthy property of the powders is size distribution, although one main point of difference between the two carbonyl nickels is in their apparent density. Another property (not tabulated) is the state of oxidation of the powder as received. Nickel has only a superficial coating of oxide whereas the other powders have all oxidized to a considerable extent. In the instance of molybdenum and cobalt, the oxygen was readily removed by heating in dry hydrogen. An appreciable drop in resistance results therefrom. The powders also differ in compactability; e.g., pure chromium cannot be compacted as received, but first must be annealed, while other metals differ in the amount of alumina which may be added and still form a compact strong enough to handle.

Powder Blending

It was decided first to see how far along the composition scale simple blending methods could be used to prepare mixtures having resistances suitable for flash sintering and later to experiment with more elaborate methods near the limits of this range. The procedure was to tumble and blend the component powders for one hour in a jar equipped with wire baffles. Other work in the powder metallurgy field had shown that a relatively uniform mixture is obtained by this method of blending. Later, other more effective methods of blending were developed, but were not used extensively since it was shown that the more effective methods did not produce any radical change in resistance for most of the compacted powders nor change the relative order of resistance of the metals investigated. Resistance measurements at 1000 lbs. are shown in Figures 11, 12, 13, and 14.

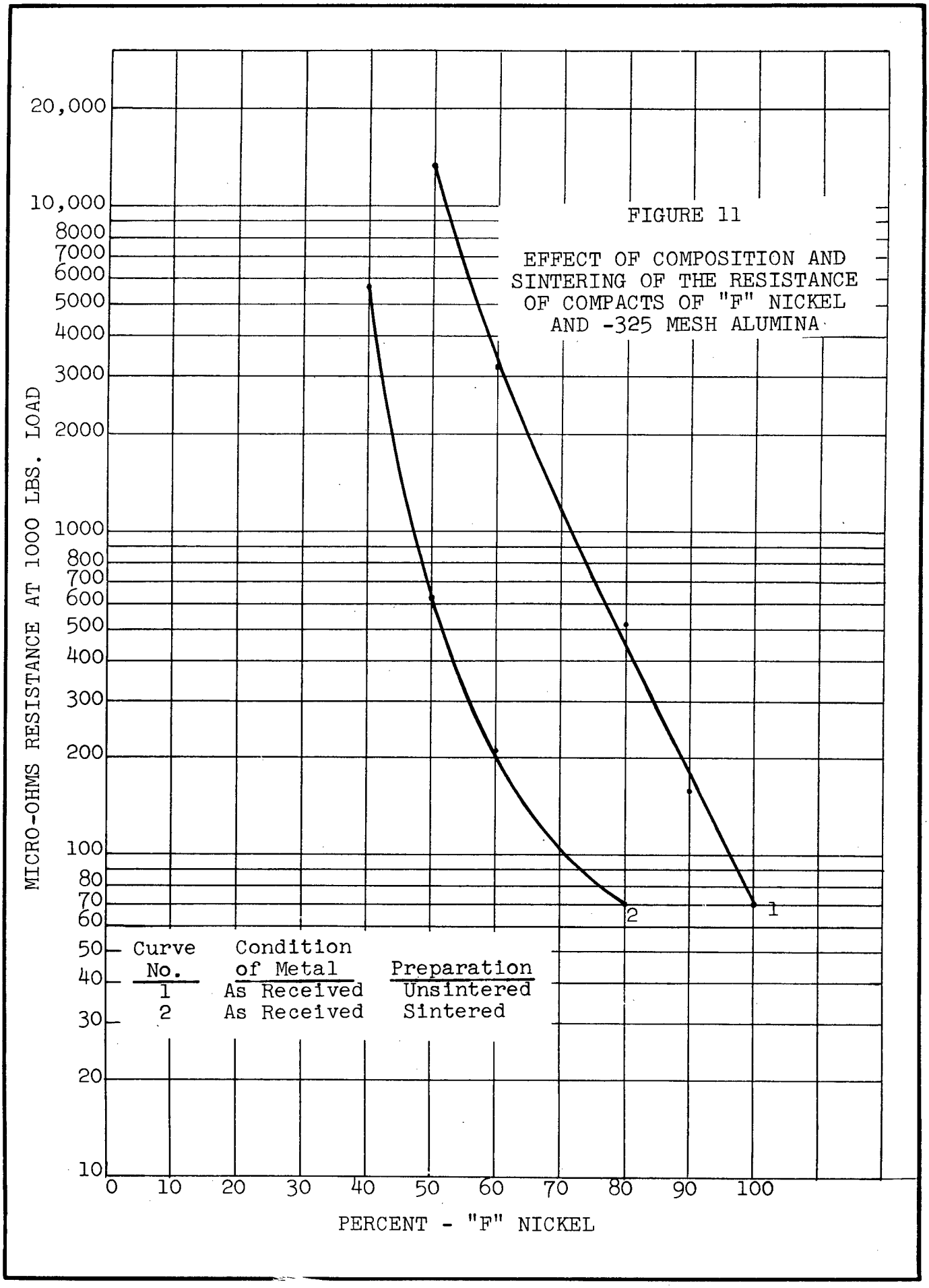


FIGURE 11
EFFECT OF COMPOSITION AND SINTERING OF THE RESISTANCE OF COMPACTS OF "F" NICKEL AND -325 MESH ALUMINA
MICRO-OHMS RESISTANCE AT 1000 LBS. LOAD
20,000
10,000
8000
7000
6000
5000
4000
3000
2000
1000
800
700
600
500
400
300
200
100
80
70
60
50
40
30
20
10
0
10
20
30
40
50
60
70
80
90
100
PERCENT - "F" NICKEL
1
2
Curve No. | Condition of Metal | Preparation
1 | As Received | Unsintered
2 | As Received | Sintered

FIGURE 12

EFFECT OF COMPOSITION AND SINTERING OF THE RESISTANCE OF COMPACTS OF "A" NICKEL AND -325 MESH ALUMINA

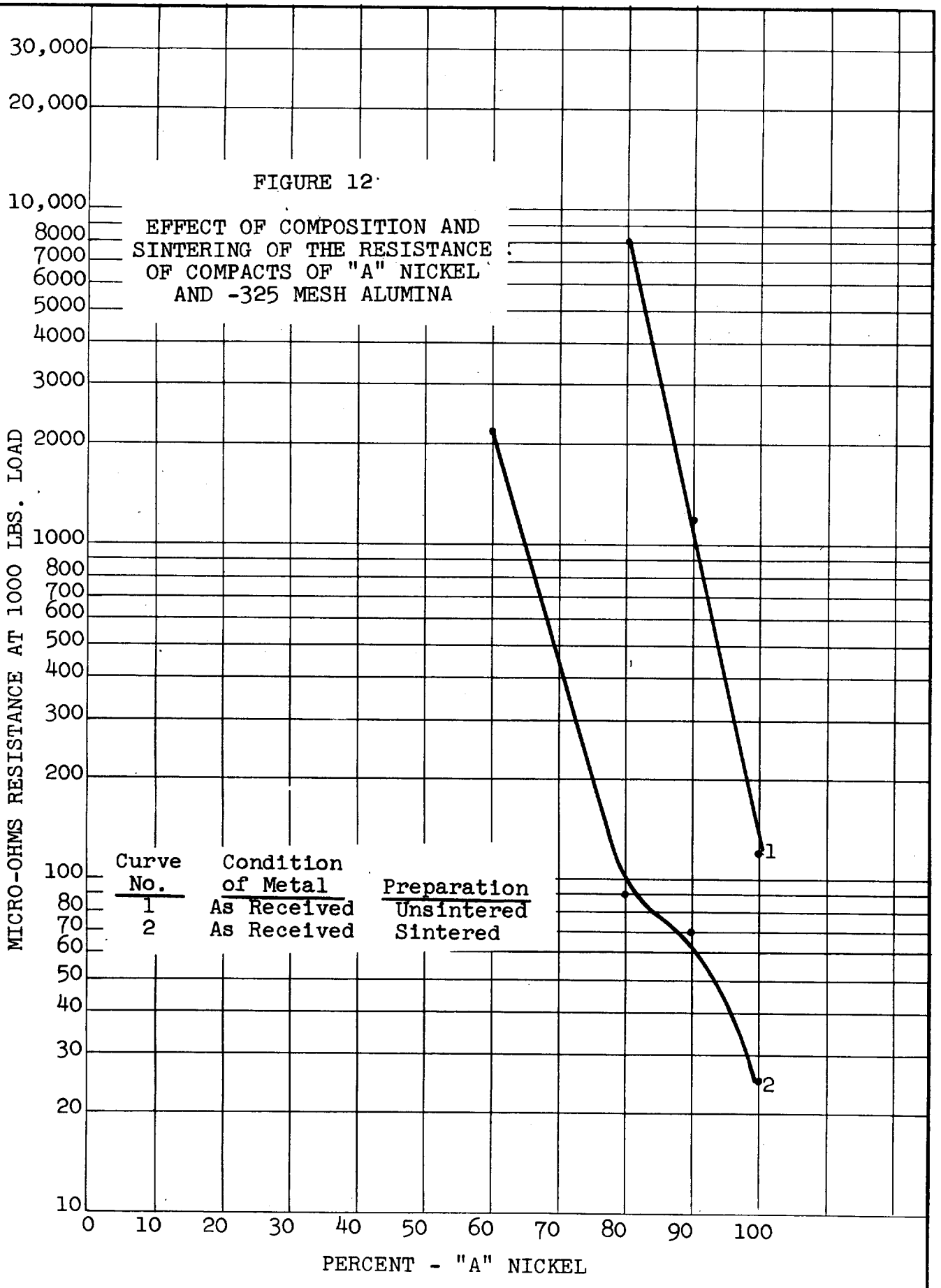


FIGURE 13

EFFECT OF COMPOSITION, SINTERING, AND REDUCTION OF POWDER ON THE RESISTANCE OF COMPACTS OF MOLYBDENUM AND -325 MESH ALUMINA

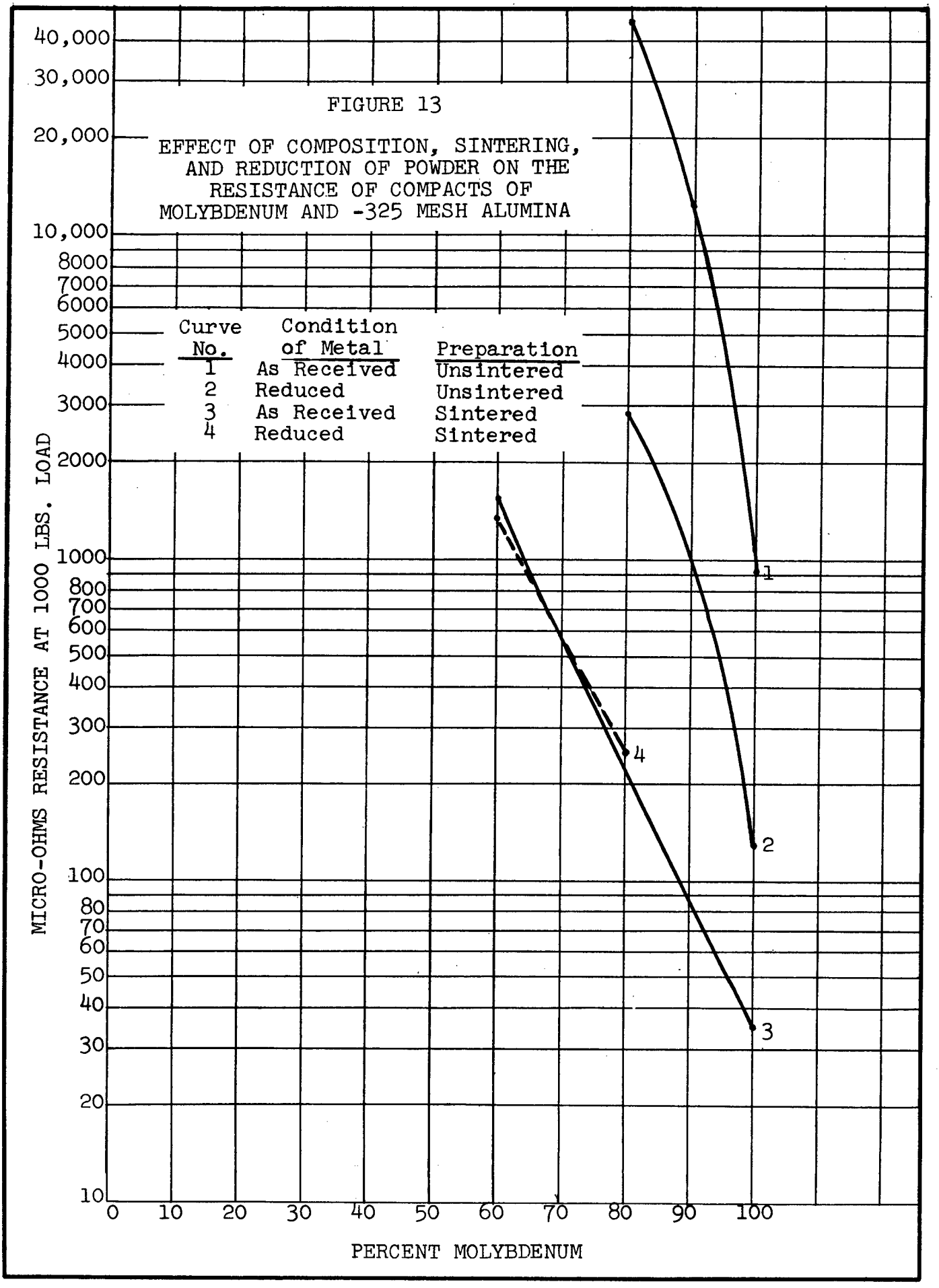
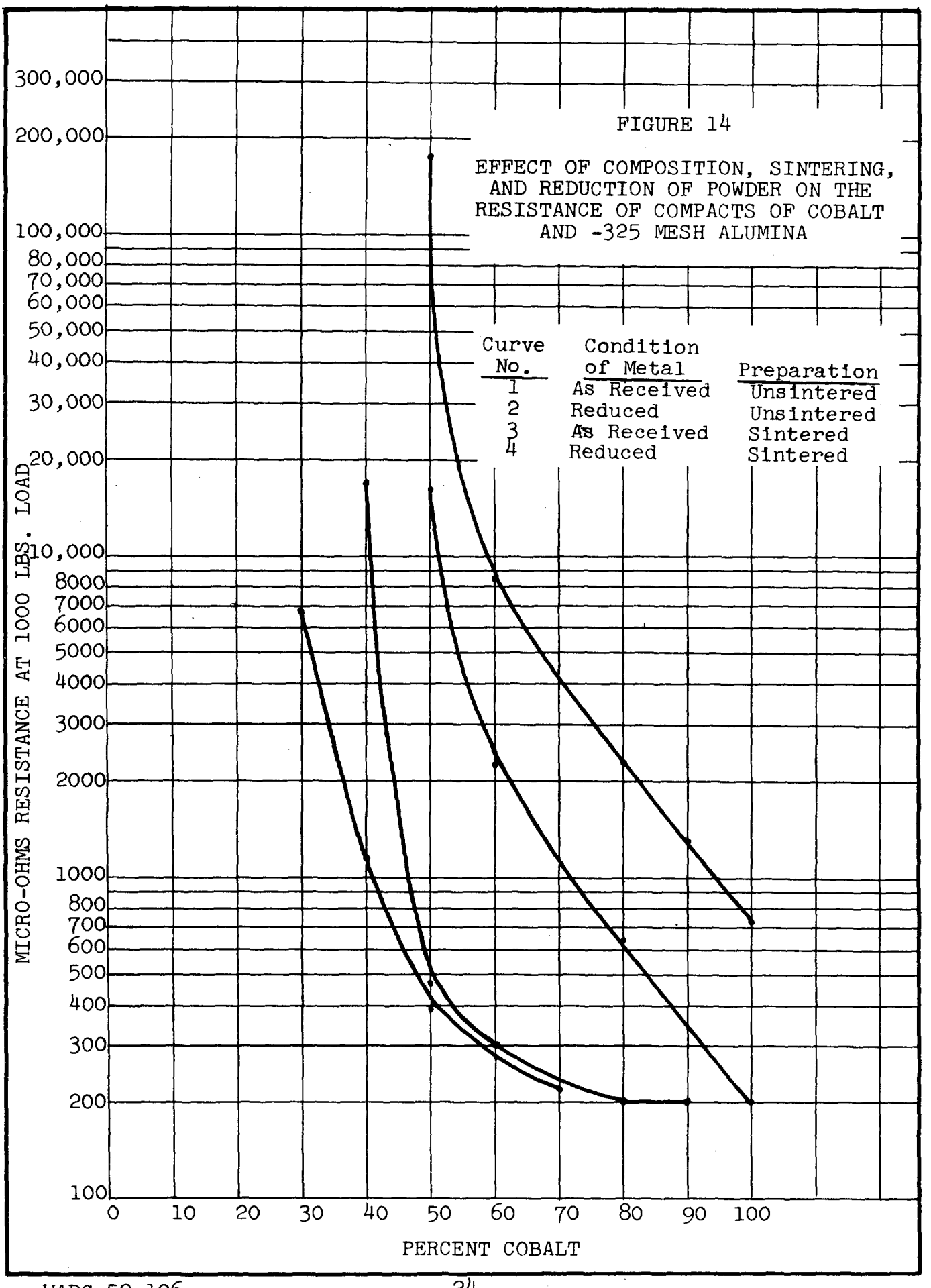


FIGURE 14

EFFECT OF COMPOSITION, SINTERING, AND REDUCTION OF POWDER ON THE RESISTANCE OF COMPACTS OF COBALT AND -325 MESH ALUMINA



Effect of Particle Size and Liquid Carrier on Resistance

Comparison of Curves 1 and 2 of Figure 11 with Curves 1 and 2 respectively of Figure 12 will serve to show the beneficial effect of greater particle surface and lower apparent density of metal powders on resistance. The "F" nickel powder is fluffy while the "A" nickel is more granular. The lower resistance of "F" nickel mixtures is due to more effective distribution of the nickel over the alumina surface. In an effort to enhance this effect, the mix was milled with ceramic balls in the proportions noted in Table II of the Appendix. Two diametrically opposite effects were observed. As shown in Figure 15, compact resistance increases with milling time apparently due to increase in relative surface of the alumina due to the effect of crushing. On the other hand, the use of carbon tetrachloride as a carrier improves the distribution of metal particles with a resultant decrease in resistance. This is most apparent when the tabulated figures for tumbling and wet stirring with a spatula are compared. The stirring method, which is quite ineffective when accomplished in the dry state, produces a lower resistance when done wet than does tumbling alone. To improve mixing beyond that possible to secure with a spatula and at the same time not produce any appreciable comminution of the alumina, mixing was carried out with 3/4" diameter maple rods, using carbon tetrachloride with the results shown in Table II of the Appendix. Of the methods tried, it was found that short time mixing produced the best results, while excessive mixing resulted in abrasion of the rods.

The blending of titanium carbide introduced a different problem. This material is received as a relatively coarse powder (Table I of the Appendix) and must be milled to small size to produce a uniform sintered product. Milling increases resistance, as may be seen in Figure 8, in which a comparison between Curve 1 for steel ball milling and Curve 2 for mixing with maple rods can be observed. The product is contaminated with iron, however, due to abrasion of the balls. The iron content was found to rise from 0.8% to about 3.8%.

E. Compact Processing

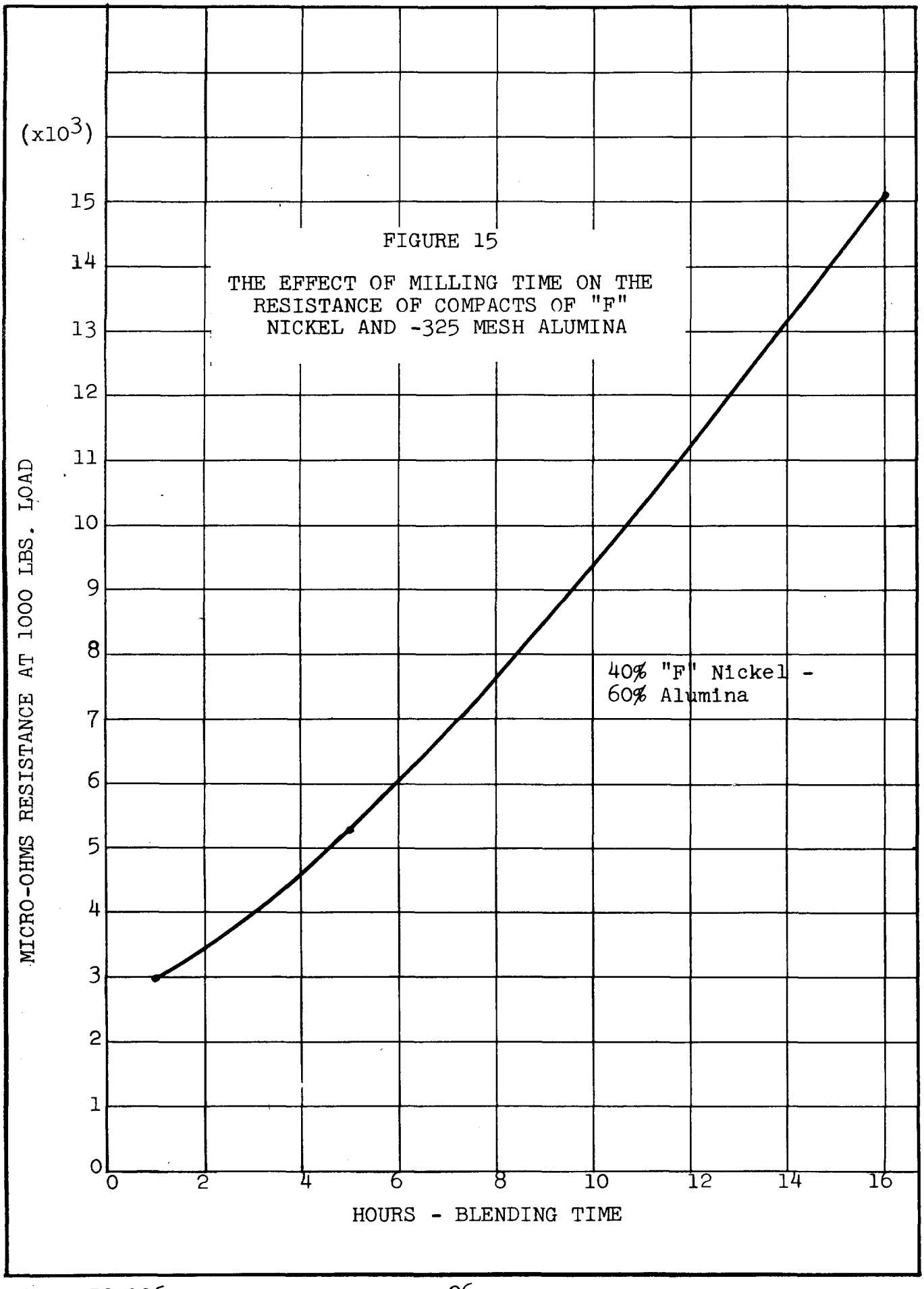
Presintering

Good blending practice will lower resistance, and further improvement is possible if the metal powders are properly processed. The beneficial effects of using powder of greater particle surface have already been noted in comparison of Figures 11 and 12. Reduction of oxides in the metal powder produces additional improvement as is shown in Figures 13 and 14 for molybdenum and cobalt respectively. However, still lower resistances result if reduction is carried out on green compacts at temperatures high enough to produce partial sintering. This may be seen by comparison of Curves 2 and 3, Figures 13 and 14. Even when reduction is not involved, this presintering is effective in lowering resistance (compare Curves 1 and 2, Figures 11 and 12, and Curves 2 and 4 of Figures 13 and 14). All presintering was conducted at 1100°C in dry hydrogen.

The resistance of presintered compacts is about the same, whether the powder is freshly reduced before presintering or not.

FIGURE 15

THE EFFECT OF MILLING TIME ON THE RESISTANCE OF COMPACTS OF "F" NICKEL AND -325 MESH ALUMINA



As shown in Figure 16, presintering accomplishes its effect quickly and perhaps independently of mixing method. It is probable that presintering reduces the resistance by removing oxide barriers and establishing many more points of metallic contact within the compact, as well as strengthening the contacts which were produced during the compacting operation. It may be significant that on loading presintered specimens well above failure, there is no great variation in resistance as might be expected if a conducting skeleton were being destroyed.

Compact Density

A fairly obvious method of reducing resistance is to press the green compacts to the highest feasible pressure before presintering. Figure 17 shows that presintered compacts of high green density have lower resistance, although the relationship between density and compacting pressure is not in direct proportion. The pressure of 50 tsi, used in most of the current work, is the highest at which reasonable die performance can be expected.

Application of Conducting Salt Solutions to Powder Compacts

The preceeding techniques seem adequate to extend the range of sinterable compositions to as low as 30% metal in the case of cobalt, but for higher resistance materials, special treatments were considered. It was felt that if metal-ceramic compacts were saturated with conducting solutions of salts, it might be possible to pass enough current to initiate heating. Accordingly, the resistance of saturated solutions of aluminum nitrate, chromium nitrate, cobalt nitrate, and nickel nitrate were measured and found to be 10, 14, 12.5, and 13 ohm-centimeters. Even solid nickel nitrate ($Ni(NO_3)_2.6H_2O$) compacted at 50 tsi had a resistance of 5 ohm-centimeters. Consequently, since published tables 2/ promised no low resistance values for other salts, this line of investigation was carried no further in the present program.

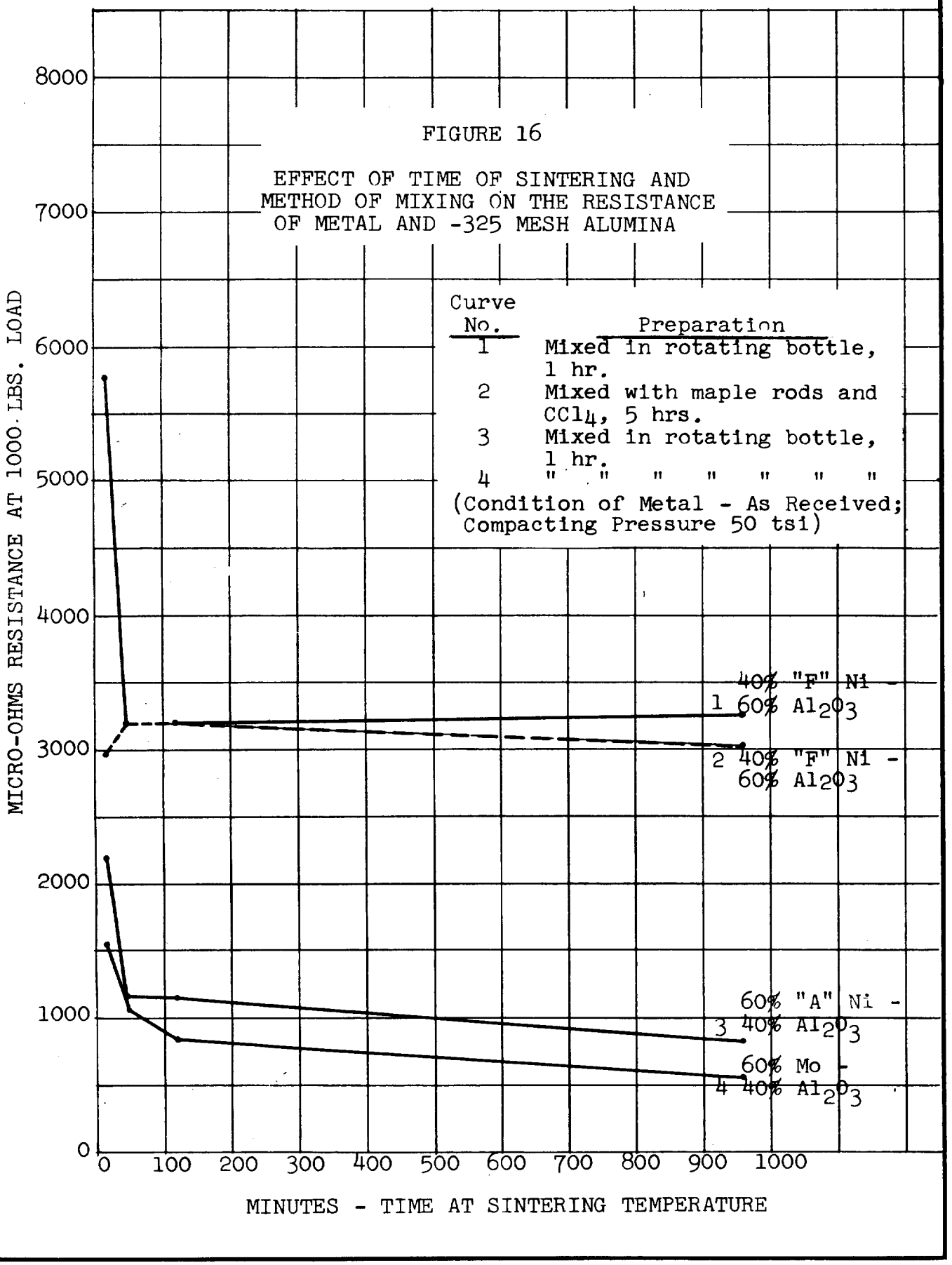
Deposition of Metal Binders

Another possible procedure was to wash the non-conductor with a metal salt solution which was then evaporated and the metal salt changed to oxide before final reduction to its elemental state. This method was expected to produce more uniform distribution of the metal. Using molybdic acid and -325 mesh alumina, 4.96% molybdenum metal was precipitated. Using nickel nitrate, 3.78% nickel was precipitated. In neither case did these low metal compositions have a usable resistance. When metal powders were added to bring the compositions to 60 and 40% metal respectively, the resistances of compacts prepared therefrom were much higher than those made from mixtures prepared by conventional blending techniques.

2/Perry-Chemical Engineers Handbook, 2nd Edition (1941)

FIGURE 16

EFFECT OF TIME OF SINTERING AND METHOD OF MIXING ON THE RESISTANCE OF METAL AND -325 MESH ALUMINA



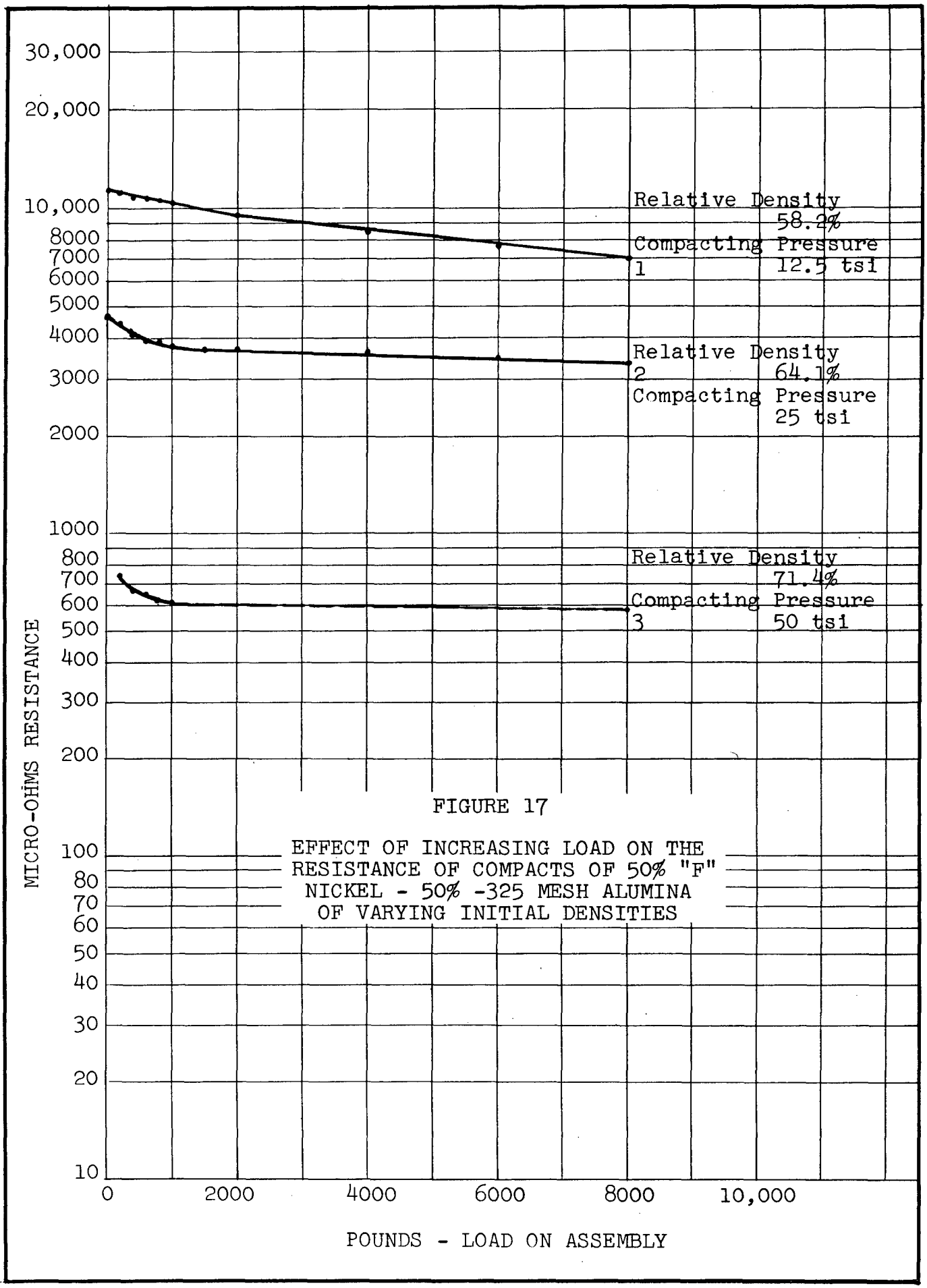


FIGURE 17

EFFECT OF INCREASING LOAD ON THE RESISTANCE OF COMPACTS OF 50% "F" NICKEL - 50% -325 MESH ALUMINA OF VARYING INITIAL DENSITIES

Co-precipitation of Hydroxides

Another method of securing an intimate mixture which would readily form a ceramet was co-precipitation of nickel and aluminum hydroxides from the nitrates, followed by conversion to oxides and the subsequent reduction of the nickel oxide at 1600°F for one hour in hydrogen to metal. This was tried with controlled quantities of nitrate solution to yield a 40% nickel - 60% alumina mix. When this mixture was compacted and the compacts given a presintering treatment, their resistance was about 90,000 micro-ohms, which is well above the value for sintered "F" nickel-alumina of the same composition.

SECTION IV

SINTERING

A. General Considerations and Findings

Compact resistance and equipment have been discussed. Compacts which have been flash sintered must have high density to be well sintered; however, this requirement does not by itself assure good strength. It must be established:

1. That compacts have appropriate initial resistance to permit satisfactory sintering.

2. If there is a critical set of sintering conditions which produce optimum and reproducible properties in a specific instance.

Sintering properties of powder mixtures were established by sintering a wide variety of compositions. While time restricted the number of experiments, sufficient data have been accumulated to indicate that compacts of (a) metals, (b) metal-carbide combinations, and (c) metal-refractory ceramic combinations can be sintered without difficulty.

Nickel compacts were sintered to theoretical density, in the initial stages of the project, to ascertain the operational characteristics of the machine. 50% nickel - 50% alumina compacts with a specific resistance before sintering of about 600 micro-ohm centimeters were easily sintered to 98% of theoretical density. Compacts made with cobalt and molybdenum were sintered with equal facility even when resistances were considerably higher. Compacts of 80% titanium carbide - 20% nickel were readily sintered, although their specific resistance was of the order of 700 micro-ohm centimeters.

B. Nickel-Alumina Compacts

Presintering

Preliminary work on 50% nickel - 50% alumina developed information shown in Figure 18 and Table III of the Appendix illustrating the effect of varying sintering conditions. It is observed that if heat setting is plotted against pulse duration, the points for the successfully sintered specimens fall within a narrow band, quadratic in shape. This relation is better defined for the presintered specimens than for the non-presintered ones. The densities, appearance, and cross-sectional uniformity of the presintered specimens is superior to those which were not presintered. Superiority of presintered specimens holds also in the instance of titanium carbide-nickel mixture. Consequently, the titanium carbide specimens were presintered for 60 minutes at 1100°C, unless otherwise noted. It is assumed, as a characteristic of the quadratic shape of the curve, that the following equation obtains:

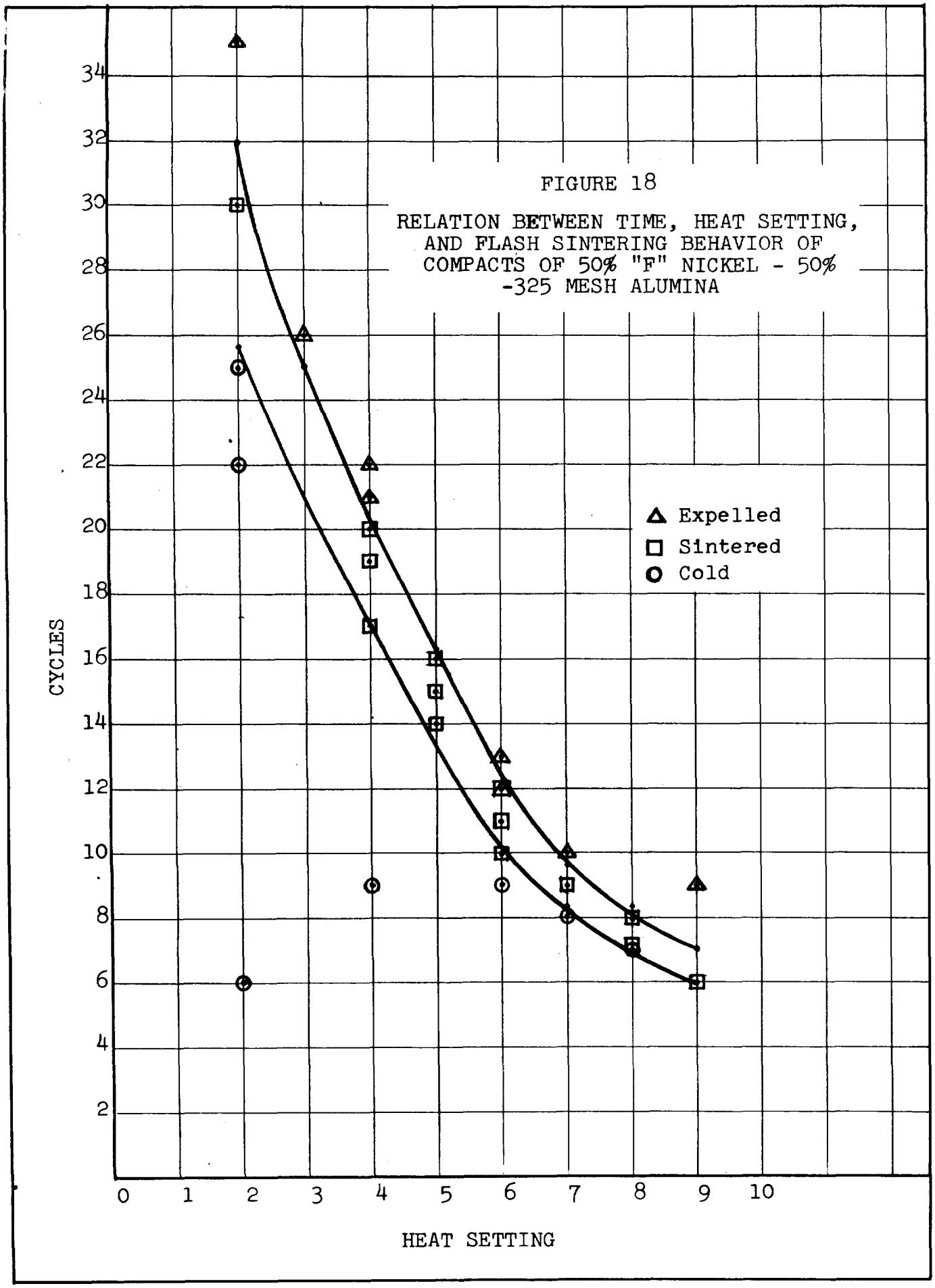


FIGURE 18
RELATION BETWEEN TIME, HEAT SETTING, AND FLASH SINTERING BEHAVIOR OF COMPACTS OF 50% "F" NICKEL - 50% -325 MESH ALUMINA
Expelled
Sintered
Cold
CYCLES
HEAT SETTING
34
32
30
28
26
24
22
20
18
16
14
12
10
8
6
4
2
0
1
2
3
4
5
6
7
8
9
10

$H^n R t = K$

where n is in the order of 2 and

H = heat setting

R = initial resistance

t = number of cycles in pulse

K = constant for compacts of identical prior history

This relationship is less well followed in the case of the titanium carbide - nickel, but does serve as a rough guide to set controls.

Experiments were conducted with nickel-alumina compacts to study the effect of pressure during sintering. It was found that the heat setting for sintering could be lowered at higher pressures. It was demonstrated that within the parameters explored, settings resulting in equal energy input produced equivalent results. The relative contribution of pressure to total energy was estimated from its effect on the resistance of compacts. Pressure had the same effect on the carbide compositions.

Control and Processing Variables

The variety of available controllable sintering conditions is great. This may be appreciated from the following tabulation:

MACHINE VARIABLES

	Pulse 1	Interval	Pulse 2	Load Holding
Time	*	*	*	*
Heat Setting	*		*	
Load	*		*	

Other variables, introduced in the preparation of the specimen prior to sintering, include compact geometry and mixing or milling, pressing, and presintering conditions.

C. Titanium Carbide - Nickel Compacts

Elements Affecting Control of Sintering Programming

Experiments with 80% titanium carbide - 20% nickel compacts disclosed that at almost any load and pulse sequence, a heat and time combination could be chosen to produce effective sintering, with the following reservations:

1. When a single pulse is used, the current rises exponentially with time; consequently, the last cycle adds a quantity of heat as great as several of the early cycles. This may cause a lack of heat control which can be overcome by substituting for the last cycle several cycles of a closely following pulse set at a much lower heat.

2. Long compacts tend to heat more in the center than at the ends in a single pulse. It has been possible to minimize this non-uniformity by the use of a double pulse.

3. Limitations imposed by the construction of the equipment and the pressure regulating system make accurate work at extremes of pressure difficult.

4. Most specimens initially were 2.16" long by 1/2" diameter. During sintering, the length was reduced to about one-half original size. A piece having a large surface area in contact with the die walls, may exhibit substantially different sintering characteristics from those of a piece much shorter in height, since the long piece must slip past much greater wall area as it becomes densified.

The results of 18 different sets of conditions are summarized in Table A and are presented in detail in Table IV of the Appendix. These results support the view that there is no single optimum set of conditions. This may be attributed to one or more of the following reasons:

1. It is possible that stresses or cracks introduced during cooling after sintering weaken what would otherwise be good compacts.

2. Unidentified factors concerned with the preparation of the powder may affect consistent sintering to high strength.

3. The geometry of the specimen may preclude proper consolidation, for even though many flash sintered compacts apparently have a high density, this property is not necessarily reflected in the microstructure or transverse rupture values. This has been noticed especially when the compacts are long with respect to their diameter.

It was observed early in this investigation that occasionally, when sintered under conditions assumed to be identical, one specimen sintered perfectly, a second specimen sintered only partially, while a third specimen became so plastic that material extruded around the wafer. This illustrates the difficulty of correlating sintering conditions with initial resistance. Likewise, there was found to be no correlation of sintering conditions with final hardness or density. The relative peak currents, cycle by cycle, are shown in Figure 19 for a set of supposedly identical specimens.

Review of Operations

Possible causes for the lack of reproducibility in the instance of titanium carbide - nickel compositions were studied by reviewing and checking the entire sequence of compact preparation and flash sintering operations, which are outlined below:

TABLE A

SINTERING CONDITIONS AND BEHAVIOR OF SPECIMENS PREPARED FROM 80% TITANIUM CARBIDE - 20% NICKEL

These specimens were made by sintering compacts prepared as described in the text, under Review of Operations. For details concerning the various specimens, see Table IV of the Appendix.

Ram load lbs.	Pulse #1 Heat Setting	Pulse #1 Firing Time, Cycles	Pulse #2 Heat Setting	Pulse #2 Firing Time, Cycles	Interval Between Pulses, Cycles	Total Length (Green), inches	Number of Specimens in Group	General Distribution of Flash Sintering Behavior and Results Obtained
3300	4.5-6.5	38	None		-	2.1	5	All specimens in this group appeared to have been hotter in the center than at the ends. Those which seem to be well sintered at either location broke when held in a vise and tapped with a hammer.
3300	10.0	16-18	None		-	2.1	3	Specimens broke easily under impact when held in a vise. Relative densities of specimens ranged from 96% - 98%.
4100	8.6	17	None		-	2.1	4	Specimens sintered, some porosity, relative densities ranged from 97% - 98%.
4100	3.0-4.0	16	8.0-8.6	15	210	2.1	3	All specimens bulged in center. Modulus of rupture value on bar machined from one specimen was 75,000 psi.
4100	8.0-8.9	16	2.8-6.0	15	210	2.1	6	Center of specimens generally appeared to have been hotter than the ends. Modulus of rupture values ranged from 30,000 to 134,000 psi.
4100	9.4-9.6	16	3.8-5.0	15	210	1.5	12	Some specimens bulged in center and many broke under impact when held in a vise. Relative density was generally over 99%.
4000 Pulse #1 8000 Pulse #2	4.8	15	6.4-7.8	10	2	2.1	27	Seven specimens were brittle, five were extruded, seven were incompletely sintered, ten gave modulus of rupture values ranging from 32,000 to 63,000 psi. The center density of the specimens (density of the rupture bars) was generally about the same as the overall density of the original specimens. These specimens were flash sintered in four successive days; those sintered on the first two days were uniformly poor.
6200	2.0-10.0	6-29	None		-	0.7	27	Most of these specimens had heat and time so balanced that they sintered, but in every case a crack, transverse to the axis, started in the center of the compact and split it in two halves.
6200	3.5	15	4.0-6.0	18	186	2.1	21	Eight specimens were brittle, two were expelled, three were incompletely sintered, eight gave modulus of rupture values ranging from 48,000 to 118,000 psi. Relative density was over 98% for the rupture bars; over 99% for the original specimens.
6200	3.5	16	6.8-6.9	15	210	2.1	5	Two specimens were incompletely sintered; three appeared to be well sintered giving modulus of rupture values ranging from 80,000 to 173,000 psi.
6200	6.6-7.7	15	3.5	16	222	2.1	31	Seven specimens were broken when removed from die holder, two were expelled violently, eleven had extruded material, eight gave modulus of rupture values ranging from 38,000 to 67,000 psi, the balance were incompletely sintered. Relative density of sintered specimens usually was over 97%.
6200	6.5-6.9	16	3.5	15	210	2.1	6	One specimen was expelled, one was incompletely sintered, the ends of most of the others were upset. Modulus of rupture values ranged from 44,000 to 72,000 psi. Relative density was about 98.8% for rupture bars and about 99.8% for original specimens.
6200	5.0	19	6.5-9.0	6	1	2.1	19	Two specimens were brittle, three were expelled, nine were incompletely sintered, six gave modulus of rupture values ranging from 38,000 to 121,000 psi. Relative density was over 98% for rupture bars; over 99% for original specimens.
7900	4.2	15	5.6-6.4	10	1	2.1	12	Almost all specimens appeared to be sintered; four broke easily under impact when held in a vise, eight gave modulus of rupture values ranging from 56,000 to 99,000 psi. Relative density was about 98% for rupture bars and 99% for original specimens.
7900	6.0	16	2.6-3.6	10	1	2.1	7	Five specimens broke on removal from die holder or under impact when held in a vise; three specimens were extruded.
12,000	4.0-4.5	38	None		-	2.1	3	Specimens appeared to have been hotter in center than at ends and broke easily under impact when held in a vise.

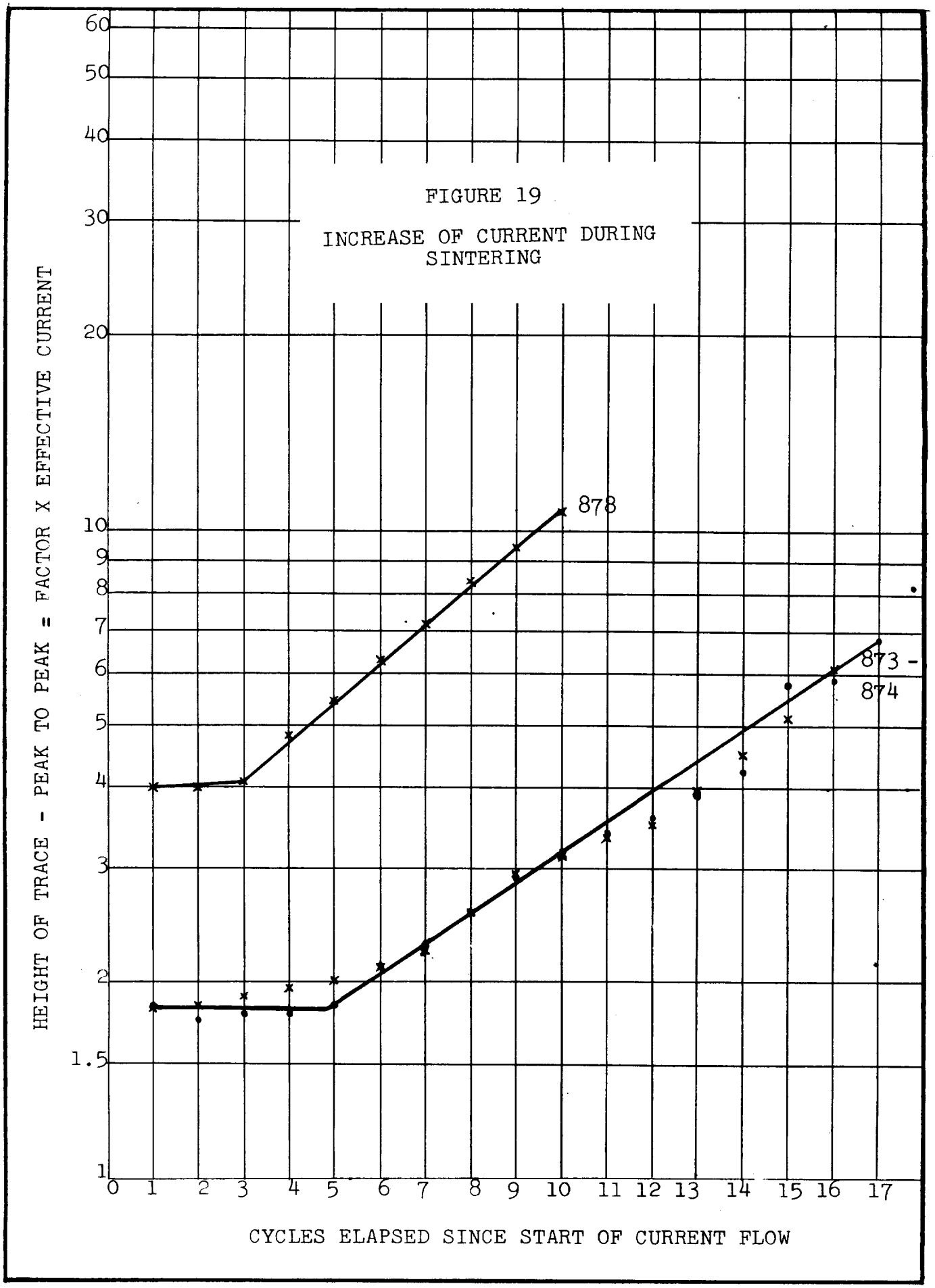


FIGURE 19

INCREASE OF CURRENT DURING SINTERING

1. Powders

Powders, as received, are stored in closed containers after having been checked for their physical and chemical properties and screen analysis. (To date the powders have been used in the particle size as received; future work will include the testing of powders which have been separated into size fractions.) In the current program, two samples of titanium carbide, of the analyses shown in Table I of the Appendix, and one lot of carbonyl nickel (battery type), also described in Table I, have been used.

2. Milling

A mixture of 240 gms. of titanium carbide, 60 gms. of nickel, and 3 gms. of paraffin (dissolved in 60 ml. of carbon tetrachloride) was placed in a 5" long by 5" diameter steel ball mill with 3 pounds of 5/8" diameter steel balls. The mixture was milled for a period of 24 hours. At the end of each 8 hour interval, additional carbon tetrachloride was added to replace that which evaporated during milling. When two batches were milled at the same time, they were blended together in the same ball mill for one hour additional to insure homogeneity.

(a) In the initial work, it was found that the rate of evaporation of carbon tetrachloride was somewhat critical. To maintain the evaporation rate constant, precautions were taken with the gasketting of the mills and the mixture was milled to a slurry of uniformly thin consistency.

(b) As stated above, steel balls 5/8" diameter were used for all milling; these balls were rejected when their size became reduced to 1/2" in diameter. No examination of the uniformity of size and distribution of carbide and nickel in the powder mix was made, but based on an examination of the sintered compacts and the experience with other carbide materials, it was assumed that the distribution would be uniform.

3. Drying of Milled Mix

After milling, the powder and steel balls were poured from the steel jar mill through a 1/4" screen; the powder adhering to the mill and steel balls was scraped off and added to the slurry which was evaporated at 40°C, leaving a residue of completely dry powder. The dried powder cake was then broken into a fine powder, screened and sieved for use in the preparation of test specimens.

4. Compacting

The powder was pressed into 1/2" diameter compacts of selected lengths at a pressure of 50 tsi, using double action pressing. A steel die, lubricated with calcium stearate in carbon tetrachloride solution, was used for the pressing operation. It was necessary for the powder to be completely dry to prevent sticking of the punches to the top and bottom surface of the compacts. The compacted material was so fragile that compacts pressed from dry powder tended to crumble under normal handling conditions. Presintered compacts have greater strength and are thus much more desirable from this standpoint.

5. Presintering

Compacts were presintered in a closed carbon boat in a hydrogen atmosphere tube furnace. They were first preheated for 20 minutes at a temperature of the order of 300 to 400°C to volatilize the paraffin. Compacts were then presintered at 1100°C in hydrogen for one hour. After this, the carbon boat and compacts were cooled to room temperature in a water jacketted cooling chamber under a hydrogen atmosphere for 20 minutes.

(a) Time and temperature values were chosen as a result of experience with comparable materials. Table IV of the Appendix shows the effect of a 5 hour presinter compared with 1 hour. Time and temperature relationships were held sufficiently close so that under comparable conditions no appreciable difference in resistance or flash sintering behavior was noted between different boat loads of presintered compacts. The advisability of using carbon tetrachloride in the furnace gas to eliminate titanium oxides is suggested by the work of Fattinger 3/, but this was not explored.

(b) It appears that the resistance drop which results from presintering is produced by the formation of a more conductive skeleton material in the compact rather than an improvement in the coating of carbide particles by nickel. Before the advent of presintering, green pressed compacts were chosen for the early work in preference to loose powder, as the use of loose powder necessitates the employment of an excessively long liner and sintering machine stroke; also the

3/Kingston - "Physics of Powder Metallurgy" - Pg. 297

problem of securing uniform compaction is presented since a liner makes a poor compacting die. Resistance also increases because of the lesser degree of compaction obtainable under the lower loads of the flash sintering machine

6. Resistance Measurement

Resistance was measured in the sintering machine by loading the compacts in the die assembly and closing the movable head of the machine on a contact insulated from the head. Values obtained should be similar to those which exist just prior to initial passage of current during the flash sintering cycle.

(a) The resistance of a considerable number of compact assemblies are shown in Figure 5. Since an appreciable spread in values was found, studies were made of possible sources of variation which might occur in the determination of resistance. However, only the tests described below, which were conducted in the final month of the period covered by this report, were fruitful in eliminating these variations. These tests consisted of check determinations on both the hydraulic testing machine and the flash sintering machine, which were run as carefully as possible to duplicate conditions of loading and die assembly set up. It had been observed that the presintered compacts had low shear strength; consequently, it was thought that they might be fractured in a heterogeneous fashion upon application of load in the flash sintering machine inasmuch as air pressure on the ram was applied suddenly in normal practice. Therefore, to avoid such sudden impact loading, the movable head of the machine was first brought to rest gently on the compact in the die assembly, and then the pneumatic pressure was bled gradually into the upper piston chamber to increase ram load to the desired level. Compacts of different lengths were employed in these tests. The average resistance values at various loads for each length are plotted in Figure 9 which reveals practically no difference in resistance (for a given load and specimen length) between the flash sintering machine and the hydraulic testing machine. Moreover, the duplication of results among the three identical specimens used for each length was excellent, the spread of values being narrow and

approximately the same for each machine.

(b) When a load was reapplied after having been once removed, the resistance was higher for the second application than it was initially. For this reason, care has been exercised to load each specimen only twice, once to measure resistance and once to sinter.

7. Compact and Liner Behavior

In the performance of both resistance measurement and flash sintering operations, presintered compacts were compressed by the electrode plungers within ceramic liners of Al-Si-Mag 35 composition. It was noticed that compacts above one-half inch long would fail in shear in most instances under 1000 lb. load and that the liner usually cracked by mechanical failure, due to its internal and external eccentricity. Both of these effects may independently or in combination be responsible for some of the remaining occurences of variables in resistance measurements. In flash sintering, however, another parameter, namely a drastic and instantaneous temperature rise, is introduced which intensifies liner failure because of the severe thermal shock conditions imposed thereby. In the selection initially of refractory insulating die liners, consideration was given to obtaining commercially available materials having good thermal shock resistance (low coefficient of expansion), dense or impermeable surface, high softening temperature, and reasonably good dimensional tolerances. Certain Al-Si-Mag ceramics were adopted because previous experience indicated they would meet these requirements more closely than other materials known at the time. While Al-Si-Mag 202 possessed most of the foregoing characteristics, including good thermal shock resistance, its surface was found to be too porous for use in flash sintering. Al-Si-Mag 35, on the other hand, exhibited poor thermal shock resistance, having a high coefficient of expansion (8.7×10^{-6} per °C), but was extremely hard and dense. It was not thought that the poor thermal shock characteristics and the attendant cracking problem would have a bearing on achieving initial contractual objectives, but would become a limiting factor only at such time when parts were to be produced to exact dimensions. Consequently, practically all flash sintering experiments were conducted using Al-Si-Mag 35 die liners. However, breakage of the liners

now appears to be more significant as it seems to be one of the unexplored causes for the present non-uniformity in flash sintering conditions and possibly for the lack of reproducibility of physical properties and microstructure in the resultant flash sintered materials. With regard to further investigations of die liners of the refractory type, it is believed that solution of the breakage problem will not be achieved unless a suitable material of low coefficient of expansion and high softening point is found.

8. Current Changes and Head Motion During Sintering

When the current-pressure cycle is initiated, the current at any moment is a function of the instantaneous resistance. The relations during sintering may be shown by the following diagram:

$$\left.\begin{array}{l}\left.\begin{array}{r}\text{Pressure}\\ \text{Elapsed Time}\\ \text{Chemical and physical properties of compact}\\ \text{Mechanical functioning of equipment}\end{array}\right\}\ \text{Degree of Compaction}\\ \\ \text{Total energy input to "t" - heat lost to wafers and liner}\\ \hline \text{Specific heat of compact}\end{array}\right\}\ \text{Current at moment "t"} = \text{Instantaneous Temperature}$$

(a) The manner in which the current increases is shown in Figure 19 in which the peak currents of the cycles of each of several pulses are plotted. A simple proportionality factor would convert these peak values to effective current values. It will be observed that on the semi-logarithmic paper the relation is approximately linear. In effect, then, over the greater part of the sintering pulse the material can be considered as having a negative coefficient of resistance which is believed to change markedly only at the point of actual sintering.

(b) Before sintering, the specimen is compressed by the downward moving head until it will carry the initial load. The head remains nearly stationary until the compact softens and it then moves suddenly downward to approximately the final position. If the material is extruded between

wafer and liner, the head drops still further as the extrusion occurs.

Examination and Test of Flash Sintered Compacts

1. General Observations

After sintering, the liner was broken from the specimen. A light "Dixonac" (graphite suspension) wash on the liners and wafers permitted easy separation. When sintering conditions were properly chosen, the resulting specimens were of uniform dimension and smooth. If the heat was too low, the ends and periphery of the specimens were soft. If the heat was excessive, the surfaces were rough, and molten metal was occasionally expelled from the liner assembly. There was generally a soft surface layer of material from which the heat had been withdrawn by the liner during sintering. When insulating coatings were used on metal liners, this layer was found to be as much as 1/8" deep, but with ceramic liners and equal energy inputs, the layer was only a few thousandths of an inch deep.

2. Physical Tests

Specimens were prepared for test measurements by grinding off surface imperfections. Conventional Rockwell A hardness measurements were made and density determined by displacement with the results shown in Table IV of the Appendix. Numerous specimens were ground to a size suitable for the determination of the modulus of rupture. For this 1/4" x 3/8" x 1-1/4" specimens, having carefully ground surfaces, were tested as a simple beam 1/4" thick and 5/8" span. These tests indicated that good properties (specimens 660 and 623) can be obtained over the whole length of the specimen although it is difficult to reproduce properties from one specimen to the next. Values of strength, hardness, and density which were determined on the machined specimens also are tabulated in Table IV of the Appendix.

3. Metallography

Metallographic examination was conducted on a number of the modulus specimens. Polishing was difficult in the instance of the poorer specimens because they contained many areas from which constituents were readily removed. Typical good and bad structures are shown in Figures 20 and 21. Examination under oblique illumination shows the dark areas to be holes, either originally present as pores,

FIGURE 20

Photomicrograph of 80% Titanium Carbide - 20% Nickel. Flash Sintered (Sample No. 660). High Modulus of Rupture - Carbide Particles uniformly distributed in nickel matrix.

Etched with 10% Nital followed by 45% Ammonium Bisulfite solution

(Magnification 100 X)

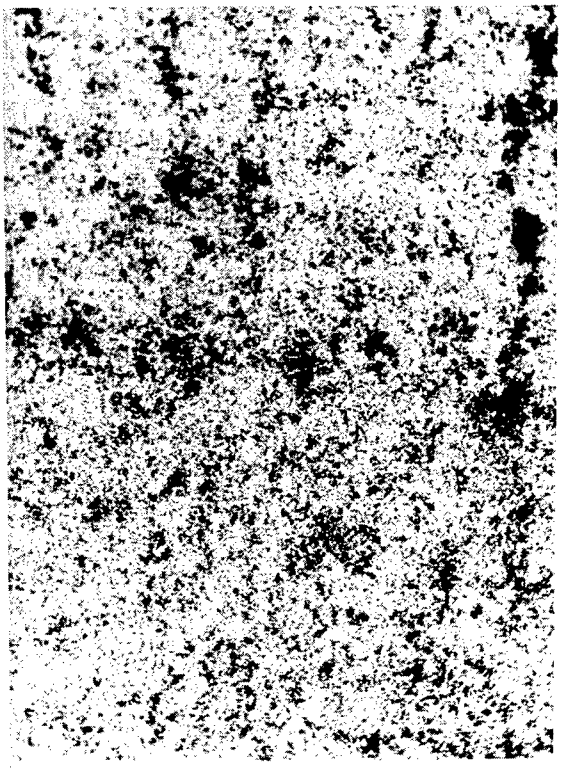


FIGURE 21

Photomicrograph of 80% Titanium Carbide - 20% Nickel. Flash Sintered (Sample No. 773). Low Modulus of Rupture - Carbide Particles non-uniformly distributed.

Etched with 10% Nital followed by 45% Ammonium Bisulfite solution

(Magnification 100 X)

or probably produced by removal of titanium carbide particles on polishing. Since examination of the fractured surfaces under magnification fails to show holes, it is probable that there are few pores in the specimens. Comparison of the densities of modulus specimens and the original flash sintered cylinders from which they were machined (see Table IV of the Appendix) indicates that with a constant sintering load, the center density is lower than that of the original specimen. In Figures 22 and 23 the structure is shown at higher magnification. The structure appears to be formed of a mass of hard polygonal particles bound together by a much smaller total amount of matrix. A core in the polygonal grains appears to be different than the outer layers (Figure 22, just discernable). Very few of the polygonal grains have assumed the rectangular shape characteristic of titanium carbide in conventionally sintered materials. In the poorer specimen, clusters of very small particles are grouped about an area which has polished as a hole, but originally may have contained unsintered material. Examination of a specimen sintered for 7 cycles at 12,000 lb. load showed fine non-polygonal carbides well dispersed in a matrix.

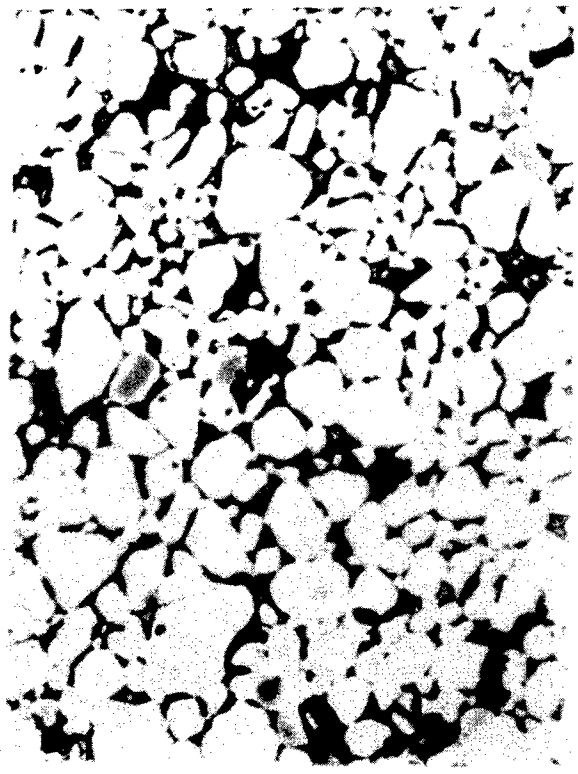


FIGURE 22

Photomicrograph of 80% Titanium Carbide - 20% Nickel. Flash Sintered (Sample No. 660). High Modulus of Rupture - Carbide Particles uniformly distributed in nickel matrix.

Etched with 10% Nital followed by 45% Ammonium Bisulfite solution

(Magnification 1000 X)

FIGURE 23

Photomicrograph of 80% Titanium Carbide - 20% Nickel. Flash Sintered (Sample No. 773). Low Modulus of Rupture - Carbide Particles non-uniformly distributed.

Etched with 10% Nital followed by 45% Ammonium Bisulfite solution

(Magnification 1000 X)

SECTION V

CONCLUSIONS AND RECOMMENDATIONS

A. Conclusions

The broad objective of this investigation has been to develop flash sintering as a means of producing temperature resistant components (i.e. turbo jet engine blades) to close dimension. Specific attention has been directed to obtaining information regarding the extent and areas of control necessary to produce sintered specimens from powder mixtures of titanium carbide and nickel. For purposes of appraisal, density, hardness, and modulus of rupture values were taken and the microstructure observed. It was initially, and is still, thought that the factors which must be considered for successful flash sintering will be identical for all materials. Depending upon the materials chosen, however, the extent of their contribution will have to be determined and subjected to appropriate control. Where specimens are to be produced from green or presintered non-conducting powder mixtures, an additional parameter is involved and means must be found to establish initial current flow.

Our investigations of titanium carbide - nickel mixtures have identified the factors which must be considered in flash sintering this material to a desired optimum property. Such scatter as occurs in test results (e.g. modulus of rupture) does not, from the data presented, seem to result from a single processing variable, but may well be caused by many or the interaction of but few. It has been assumed that Al-Si-Mag 35 liners, which will always crack due to thermal shock, are significantly responsible. It has been observed, however, that many presintered specimens crack when initially loaded over 2000 lbs. prior to passage of the sintering current. Such cracks may present areas of high resistance and thus during the sintering cycle cause metallurgical discontinuities and possibly incipient cracks adjacent to such areas. Thus it is now believed that such a condition may be of greater significance than liner failure due to thermal shock.

The many factors which have been considered in connection with the flash sintering process fall within six general categories described and appraised as follows:

1. Procurement, Preparation, and Mixing of Powder prior to Green Compacting or Green Compacting and Presintering

   It is considered that the procedures followed are adequate and are under control.

2. Green Compacting

   Optimum conditions for green compacting have been established by taking resistance measurements of compacts. It has been assumed that compacts of the same composition having close resistance values at room temperature are substantially similar. Procedures are considered satisfactory.

The following observations are considered pertinent:

(a) Compact resistance measured at room temperature decreases with increased pressure, rapidly at first then slowly.

(b) Resistance increases slowly, then rapidly as the percentage of non-conducting powder is increased in a mixture containing a conducting powder.

(c) Identical compacts will have identical resistance at room temperature only under equivalent conditions of loading.

(d) Increase in relative surface of metal powder tends to lower compact resistance.

3. Presintering (Apparatus and Control)

Presintering techniques are assumed to be adequate to provide specimens identical in quality as established by density measurements and resistance measurements taken at room temperature. It is conceivable, however, that were resistances to be taken at various increments of temperature, significant differences in quality might be observed. Also micro-hardness surveys and metallurgical examination of presintered specimens might disclose non-uniformities. Presintering for short periods of time substantially increases the conductivity of green compacts, but increasing time has little additional effect.

4. Flash Sintering (Sintering Mechanism and Control -- Processing)

The sintering mechanism is generally satisfactory insofar as mechanical and electrical components are concerned. It may be, however, that further instrumentation is warranted if closer duplication of metallurgical structure is demanded. For example, it may be desirable to have information regarding the actual loads imposed at each cycle during the current pulse. Also it may be desirable to establish the percentage of compact deformation at each cycle during the current pulse. Additional observation indicates that:

(a) Minor improvements in sintering assembly will assure more precise alignment of components during sintering.

(b) Improvements are desirable in pneumatic controls.

(c) Ram follow-up during sintering may have to be improved if such is indicated by further instrumentation.

In processing it has been observed that:

(a) Al-Si-Mag 35 liners have far too great a coefficient of expansion (8.7×10^{-6} per °C) to be resistant to thermal shock. These liners crack irrespective of the means of support employed.

(b) Al-Si-Mag 35 liners are not available within the desired dimensional tolerances, and their lack of concentricity may also contribute to poor performance.

(c) Sintering currents at low voltage have been passed through presintered powder mixtures of metal-alumina with metal content as low as 30% cobalt - 40% nickel - 60% molybdenum - 90% chromium.

(d) Special methods investigated to a limited degree to improve the conductivity of powder compacts high in non-metallic content were proven to be unsuccessful.

(e) The fact that compacts are of low enough resistance to allow the passage of a sintering current is no guarantee that they will completely sinter to a desired metallurgical structure.

(f) Equal energy inputs should produce equivalent effects on identical specimens if pressures are the same.

(g) Extension of heating time causes heat to be lost to liners and wafers.

(h) Energy may not be distributed equally over the length and diameter of a specimen.

(i) Cylindrical specimens of titanium carbide - nickel can be produced to high density and to high uniform hardness.

(j) To obtain maximum strength, densities should be over 99%.

(k) It has not been possible, as yet, to produce specimens of 80% titanium carbide - 20% nickel composition to uniformly high modulus of rupture values.

(1) Considerable latitude can be taken in the choice of pressures and currents required to flash sinter specimens to high hardness and density. As pressure is increased, the requirements for electrical energy decrease.

5. Instrumentation

Such instrumentation as has been employed was considered adequate for the initial studies undertaken. It is now apparent that further instrumentation will be necessary if the exact causes for variation in specimen performance are to be established. Means must be taken to determine resistance changes and load at each cycle during a pulse. It may also be necessary to measure compact deformation during each cycle of each pulse.

6. Methods of Inspection and Test

Such means as were taken to establish control over raw materials, mixing and blending, green compacting, presintering, and flash sintering were, until recently, considered adequate. It now appears that if the scatter in test results is to be eliminated, more must be learned about the integrity of presintered compacts. Also more attention will have to be given to the examination of metallurgical structures produced as the result of various cycle settings.

B. Recommendations

It is evident that further investigations should be initiated to establish the causes of variation experienced in specimens flash sintered from presintered compacts of 80% titanium carbide - 20% nickel mixtures. The recommended areas of investigation include the following:

1. Determination of the effect of liner cracking.

2. Determination of the effect of cracking of presintered compacts under ram pressure prior to the passage of current.

3. Determination of the ability of the mechanical and pneumatic elements of the flash sintering machine to assure adequate ram follow-up during the sintering cycle.

Further instrumentation is indicated to establish the load and resistance values at each cycle of each pulse as only by such means does it appear possible to secure the information necessary to answer the questions which remain.

It is strongly recommended that attention be given to the significance of the metallographic structures obtained and that an attempt be made to relate the structures observed with the flash sintering programming chosen.

It is suggested that after the information described in the above paragraphs have been obtained, steps be taken to investigate problems associated with the flash sintering of specimens having a geometry more comparable to the shape of turbine blades.

I

A P P E N D I X

TABLE I

PROPERTIES OF POWDERS

Powder	Electrolytic Chromium		Carbonyl Nickel		Molybdenum c.p. H_2 Reduced	Cobalt H_2 Reduced	Zirconia			Alumina		Beryllia	Titanium Carbide	
Supplier	Electrometallurgical Sales Company, Niagara Falls, New York		International Nickel Company New York, New York		Chas. Hardy, Inc., New York, New York	Adamas Carbide Corp., Harrison, New Jersey	Norton Company, Worcester, Massachusetts			Aluminum Company of America, Newark, New Jersey		Brush Beryllium Company Cleveland, Ohio	Kennametal, Inc. Latrobe, Pennsylvania	
Grade	-100 mesh	-325 mesh	7-9 microns	7-9 microns Battery type	--	--	C-30F mesh	D-8F mesh (1)	D-36F mesh (1)	48 mesh	325 mesh	325 mesh	--	--
Lot No.	33469	33484	--	--	--	--	--	--	--	NK-9938	NK-9938	--	1 16 May 1951	2 1 August 1951
Sieve Analysis Mesh Fraction														
+ 50 mesh							33.2	78.9	55.3	1.0			--	--
- 50 +100	trace	trace	3.9				23.7	19.2	28.4	33.1			0.6	0.6
-100 +150	3.3	0.6	2.5				9.1	1.7	5.3	14.8			13.0	10.8
-150 +200	12.1	1.6	4.1		trace		7.0	0.2	3.2	11.9			23.5	22.2
-200 +270	13.3	3.8	2.5		0.4		4.9		1.4	6.0			14.5	11.7
-270 +325	13.0	25.3	2.9	0.0	0.9	trace	4.0		1.2	5.2	trace	1.0	14.0	16.2
-325	58.3	68.7	84.1	100.0	98.7	100.0	18.1		5.2	28.0	100.0	99.0	34.4	38.5
Apparent Density (by Scott Volumeter) grams/cm^3	3.08	2.81	3.07	1.04	1.83	1.04	2.95	1.86	2.15	1.68	1.31	0.23	2.26	2.26
Chemical Analysis														
Chromium	99.95%	99.92%	-	-	-	-							Not detected	Not detected
Nickel	-	-	99.64%	99.69%	-	-								
Molybdenum	-	-	-	-	99.50%	-							Not detected	Not detected
Cobalt	-	-	-	-	-	97.60%							0.92	0.36
Iron	0.01	0.03	0.03	0.03	-	-							17.9	17.8
Carbon	0.02	0.03	0.11	0.10	-	-								
Oxygen	-	-	0.19	0.15	0.44	2.32								
Sulfur	-	-	0.02	0.02	-	-								
Code Designation	D	E	A	F	B	C	b	c	d	e	f	a	TiC	TiC

NOTE: (1) Contains some black particles.

TABLE II

EFFECT OF METHOD OF POWDER BLENDING ON RESISTANCE OF "F" CARBONYL NICKEL - -325 MESH ALUMINA COMPACTS

Powder Condition: As Received

Compacting Pressure: 50 tsi

Condition of Compacts: Sintered at 1100°C for 15 minutes at temperature in hydrogen atmosphere

Composition	Powder Blending Procedure Method	Time	Amp.	Load lbs.	Diameter in.	Height in.	Density g/cc	Relative Density %	Resistance MicroOhms
40% "F" Ni 60% Al_2O_3	A	1 hr.	10	1000	.507	.375	3.59	70.0	5770
	B	1 hr.		Readings over scale of meter					
	B	5 hr.		Readings over scale of meter					
	C	1 hr.	10	1000	.492	.397	3.69	71.9	3970
	C	5 hr.	5	1000	.494	.374	3.65	71.2	5280
	C	16 hr.	3	1000	.494	.376	3.61	70.4	15,100
	D	1 hr.	10	1000	.494	.379	3.63	70.8	3190
	E	15 min.	10	1000	.493	.373	3.67	71.6	4000
	F	5 hr.	10	1000	.493	.371	3.65	71.2	2970

TABLE II (Continued)

POWDER BLENDING PROCEDURES

Method Designation	Charge			Description
	Mixture gms.	Balls gms.	Solvent ml. CCl_4	
A (Standard)	50	none	none	Mixed in rotating bottle with wire to cause tumbling
B	50	453 (a)	none	Mixed in #8 Porcelain Jar Mill
C	100	100 (a)	none	" " " " "
D	100	100 (a)	70	" " " " "
E	50	none	35	Mixed with spatula in 4 oz. bottle
F	85	100 (b)	70	Mixed in #8 Porcelain Jar Mill

Notes:

(a) Ceramic Balls
(b) 3/4" x 1-1/2" Maple Rods

TABLE III

TEST RESULTS OF FLASH SINTERING OF "F" CARBONYL NICKEL AND "F" CARBONYL NICKEL - -325 MESH ALUMINA COMPACTS

Powder Mixing Procedures - Nickel Powder used as received. "F" Carbonyl Nickel - Alumina Powders blended by mixing for 5 hours in #8 jar mill with maple rods and carbon tetrachloride as the liquid carrier.

Compacting Pressure - 50 tsi

Condition of Compact - As Pressed (green)

Wafers - Stainless Steel - 5/16" thick each

Ceramic Liners - Al-Si-Mag No. 202

Load Applied for duration of firing pulse plus 10 cycles

Average Length of Compact Before Flash Sintering - .375"

Specimen No.	Composition	Heat Setting	Voltage Tap	Time (cycles)	Ram Load lbs.	Relative Density by Measurement	Behavior and Appearance
		(Single Pulse)					
344	100% "F" Carbonyl Nickel	2	1	5	2500		Slight amount of sintering. Some sticking of compact to wafers.
345		4		5			" " " " " " "
346		6		5			Slight amount of sintering. Some compression of compact.
347		8		5			Sintered. Compression of compact with pronounced sticking to wafers.
348		4		15			Substantially complete sintering producing ductile compact under hammer blow. Considerable sticking to wafers.
352	80% "F" Carbonyl Nickel - 20% -325 Mesh Alumina	4	1	6	2500	76.5	Little sintering due to insufficient heating.
353		4		8		77.1	" " " " " " "
354		4		8		76.4	Partial sintering due to insufficient heating. No sticking of compact to wafer.
351		4		10			Expulsion of some of the material.
358		4		12			Expulsion of material. Considerable sticking of flash sintered compact to wafers.
350		4		15			Expulsion of substantially all of the material.
355		5		8		81.2	Some sintering. No sticking to wafers.
357		6		8		93.5	Sintered. Some sticking of compact to wafers.
359		7		8		91.7	" " " " " " "
361		6		8	5400	94.4	" " " " " " "
360		7		8			Sintered. Considerable sticking of compact to wafers.
363		2		20	8000	95.4	Some sticking of compact to wafers.
362		6.5		8		94.5	Sintered. Some sticking of compact to wafers.
364		10		5			Violent expulsion of material.
402	50% "F" Carbonyl Nickel - 50% -325 Mesh Alumina	2	1	30	8000	79.2	Partial sintering with slight compaction. Insufficient heating.
414		6		15			Conduction at single point.
416		6		16			Expulsion of material.
415		6		17		93.4	" " " "
381		6		21			" " " "
409a		7		10		78.5	No Sintering. Slight compression of compact.
410		7		11		81.0	Little sintering due to insufficient heating. Some compression of compact.
382		7		11		79.0	Little sintering due to insufficient heating.
383-411		7		13		90.0	Appreciable sintering of compact.
412		7		14		93.6	" " " " " "
409-413		7		15			Some expulsion of material.
380		7		21			Expulsion of material
408		7		25			" " " "
446-447		8		9		91.5	Sintered.
379		8		10		89.3	" "
444		8		12			Expulsion of some of the material.
386		8		16			Expulsion of material.
378		8		21			" " " "
373		9		5			No appreciable passage of current. Insufficient heating.
376a		9		8			Some sintering. No sticking of compact to wafers.
375-377		9		9			Sintered.
376-404-407		9		10			Expulsion of material.
403		9		14			" " " "
		(Two Pulses - 2 cycles dwell between pulses)					
417		9-2	1	8-3	8000	89.1	Partial sintering. Insufficient heating.
420		9-2.5		8-10			Expulsion of material.
418		9-3		8-3		94.0	Sintered.
419		9-3		8-5			Uneven distribution of heat through compact.
401		9-9		12-12			Severe expulsion of material.

TABLE III (Continued)

TEST RESULTS OF FLASH SINTERING OF "F" CARBONYL NICKEL - -325 MESH ALUMINA COMPACTS

Powder Mixing Procedure - Blended by mixing for 5 hours in #8 jar mill with maple rods and carbon tetrachloride as the liquid carrier.

Compacting Pressure - 50 tsi

Condition of Compact - Sintered for 15 minutes at temperature of 1100°C in hydrogen

Wafers - Stainless Steel - 5/16" thick each

Ceramic Liners - Al-Si-Mag No. 202

Load Applied for duration of firing pulse plus 10 cycles

Specimen No.	Composition	Average Length of Compacts Before Flash Sintering	Heat Setting	Voltage Tap	Time (cycles)	Ram Load lbs.	Relative Density by Measurement	Behavior and Appearance
			(Single Pulse)					
475	50% "F"	.259"	5.7	1	8	8000		Sintered.
476	Carbonyl		5.8		8			" "
474	Nickel -		6		8			Mild expulsion of material.
473	50% -325		6		12			Complete expulsion of material.
421	Mesh Alumina	.375"	2	1	6			No sintering. Insufficient heating.
449			2		22			Partial sintering. Insufficient heating.
450			2		25			" " " " "
451			2		30		98.3	Compact appears substantially sintered.
452			2		35			Expulsion of material.
443			3		26			Slight expulsion of material.
370			4		9			Not sintered. Insufficient heating.
438			4		17			Sintered.
439			4		19		98.3	" "
440			4		20			" "
442			4		21			Slight expulsion of material.
441			4		22			" " " "
434			5		14			Sintered. Compact shows horizontal parting line.
437			5		15			Sintered.
436			5		15			Sintered with mild expulsion of material.
435			5		16			" " " " "
366-371			6		9			Not sintered. Insufficient heating.
430			6		10			Sintered. Compact shows horizontal parting line.
431			6		11			" " " " "
432			6		12		97.6	Sintered.
453			6		12			" "
456			6		12			Sintered with mild expulsion of material.
433			6		13			Expulsion of material.
457			6.1		12			Sintered with mild expulsion of material.
455			6.3		12			Mild expulsion of material.
454			6.5		12			" " " " "
427			7		8			Partially sintered.
429			7		9		97.8	Sintered. Compact shows horizontal parting line.
428			7		10			Violent expulsion of material.
424			8		7			Not fully sintered. Compact shows horizontal parting line.
425			8		7			Sintered.
426			8		8		94.5	Sintered with slight expulsion of material.
422			9		6		97.5	Sintered.
423			9		7			Slight expulsion of material.
365			9		9			Expulsion of material.
			(Two Pulses - 2 cycles between pulses)					
459		.375"	5.8-2	1	12-6	8000		Not sintered.
460			5.8-2		12-7			" " "
461			5.8-2.5		12-7			Sintered.
462			5.8-2.7		12-7			Mild expulsion of material.
458			5.8-3		12-7			" " " " "
			(Single Pulse)					
463		.375"	6	1	12	5400		Not sintered. Insufficient heating.
464			6.1		12			Sintered.
465			6.4		12			" "
471			6.6		12			" "
472			6.8		12			Slight expulsion of material.
466			6.4	1	12	2900		Not sintered. Insufficient heating.
467			7		12			" " " " "
469			7.5		12			Partially sintered.
470			7.7		12			Sintered.
468			8		12			Mild expulsion of material. Compact split horizontally.
477		.612"	6	1	18	8000		Not sintered.
479			6		28			Sintered. Slight expulsion of material.
481			6.2		25			Slight expulsion of material.
			(Two Pulses - 2 cycles between pulses)					
478		.612"	6-6	1	18-18	8000		Not sintered.

Note: Samples are arranged in order of increasing heat and/or time within each group.

TABLE IV

TEST RESULTS OF FLASH SINTERING OF 80% TITANIUM CARBIDE - 20% "F" CARBONYL NICKEL COMPACTS

Powder Mixing Procedure

Blended by milling 300 gram charge for 24 hours in a 5" diameter x 5" long steel ball mill using 3 pounds of 5/8" diameter steel balls with 1% added lubricant (paraffin except as noted) and carbon tetrachloride as the liquid carrier. Dried, milled powder used for preparation of compacts.

Compacting Pressure

50 tsi

Nominal Compact Diameter

1/2"

Condition of Compact

Specimens No. 643 through 654 sintered for 5 hours; all other specimens sintered for 1 hour at a temperature of 1100°C in hydrogen. Specimens No. 513 through 546 sintered in steel boat. All other specimens sintered in closed carbon boat.

Wafers

Tungsten 5/16" thick each

Ceramic Liners

Al-Si-Mag No. 35

Load Applied

For duration of firing pulse plus time indicated under hold time.

TEST RESULTS OF FLASH SINTERING OF 80% TITANIUM CARBIDE - 20% "F" CARBONYL NICKEL COMPACTS (1 HOUR PRESINTER)

Specimen Number	Nominal Length of Presintered Compacts Before Flash Sintering (in.)	Nominal Weight (gms.)	Number of Compacts Used	Ram Load (lbs.)	Resistance of Assembly (At Ram Load) (micro ohms)	Single Pulse: Voltage Tap	Heat Setting (1)	Firing Time (cycles)	Hold Time (cycles)	Density (by Displacement) (5.47 g/cc = 100%) g/cc	%	Rockwell Hardness (R_A) (2)	Behavior and Appearance
513	.390	4.76	1	8000		1	6.6	7	9	5.27	96.3		Sintered
515							6.8	7	9	5.23	95.6		Extrusion
511							7.0	7	9	4.87	89.0		"
512							7.0	7	9				"
516							7.2	7	9	5.31	97.1		Expulsion of material
514							7.4	7	9	5.34	97.6		Extrusion
529	.390	4.76	3	8000		1	7.0	14	4				Not sintered
530							7.0	18	4				Partially sintered
528							6.5	20	4				Extrusion
534							6.2	22	4				Ends sintered. Center only partially sintered
533							6.6	22	4				Minor extrusion. Center only partially sintered
532							6.8	22	4				Extrusion
531							7.0	22	4				" and expulsion of material
553a	.410	5	3	8000		2	8.0	14	4				Not sintered
555							8.6	14	4				Partially sintered
558							8.6	14	4				Ends sintered. Center only partially sintered
557							8.7	14	14				Expulsion. Center only partially sintered
556							8.8	14	4				" " " " " " " "
554							9.0	14	4				" " " " " " " "
542	.390	4.7	3	8000		7	10.0	7	4			73-83	Sintered. Center flaw on cutting. (Sectioned hardness R_A 75-87)
544						8	8.0	7	4				Partially sintered
549							8.3	7	4				Extrusion
546							8.4	7	4				Ends sintered. Center only partially sintered
548							8.4	7	4				Violent extrusion
545							8.6	7	4			75-87	Mild expulsion. (Sectioned hardness R_A 80-87)
547							8.6	7	4				Mild expulsion
543							10.0	7	4				Violent expulsion
551	.410	5.0	3	8000		8	8.0	7	4			84-87	Sintered
552							8.1	7	4			80-70	"
553							8.2	7	4				" Crack in specimen
552a							8.4	7	4				" " " "
614	.640	7.4	2	4100	3720	2	8.8	15	9				Partially sintered
617					3300		9.0	15	9				Extrusion and expulsion of material
618					3600		9.0	15	9	5.41	98.9	83-86	Sintered
619					3520		9.1	15	9	5.27	96.3	85-87	Blistered. Shrunken in diameter
615					3600		9.4	15	9	5.37	98.2	83-86	Incompletely sintered
616					3450		10.0	15	9	5.23	95.6	85-85	Sintered
628	.700	8.2	3	4100	7400	8	8.6	17	9	5.34	97.6	85-85	Sintered
629					5400		8.6	17	9	5.31	97.1	86-87	" Some porosity
631					5200		8.6	17	9				"
630					4600		8.6	19	9	5.34	97.6	86-83	Expulsion

NOTES:

(1) Samples are arranged in order of increasing heat and/or time within each group.

(2) First hardness value - center of compact; second value taken at ends.

TABLE IV (Continued)

TEST RESULTS OF FLASH SINTERING OF 80% TITANIUM CARBIDE - 20% "F" CARBONYL NICKEL COMPACTS (5 HOURS PRESINTER)

Specimen Number (2)	Nominal Length of Presintered Compacts Before Flash Sintering (in.)	Nominal Weight (gms.)	Number of Compacts Used	Ram Load (lbs.)	Resistance of Assembly (At Ram Load) (micro ohms)	Voltage Tap	1st Pulse (1) Heat Setting	1st Pulse (1) Firing Time (cycles)	Time Between Pulses secs.	2nd Pulse Heat Setting	2nd Pulse Firing Time (cycles)	Hold Time (cycles)	Behavior and Appearance
649	.720	8.4	3	4100	5800	8	3.5	16	3.5	7.5	15	9	Not sintered.
647					5800		3.5	16		8.0	15	9	Sintered. Some expulsion
650					6200		3.5	16		8.0	15	9	"
646					5400		3.5	16		8.2	15	9	" . Slight expulsion (4)
645					5300		3.5	16		8.8	15	9	"
643					6600		3.5	16		8.9	15	9	" . Some expulsion
651	.720	8.4	3	3900 (3)	5800	8	3.5	16	3.5	8.0	15	9	Sintered. Center overheated
652	.720	8.4	3	2500 (3)	8200	8	3.5	16	3.5	8.2	15	9	Not sintered. No compaction
654					7800		5.0	16		8.2	15	9	" "

NOTES:

(1) Samples are arranged in order of increasing heat and/or time of 1st. pulse within each group.

(2) Powder milled for 5 hours instead of 1 hour used before.

(3) Differential load.

(4) Transverse Rupture Specimen from Sample No. 646

Modulus of Rupture 72,700 psi
Density by Displacement
5.29 g/cc = 96.7%
Rockwell Hardness R_A 74-77

TABLE IV (Continued)

TEST RESULTS OF FLASH SINTERING OF 80% TITANIUM CARBIDE - 20% "F" CARBONYL NICKEL COMPACTS (1 HOUR PRESINTER)

Specimen Number (2)	Nominal Length of Presintered Compacts Before Flash Sintering (in.)	Nominal Weight (gms.)	Number of Compacts Used	Ram Load (lbs.)	Resistance of Assembly (At Ram Load) (micro ohms)	1st Pulse (1): Voltage Tap	1st Pulse (1): Heat Setting	1st Pulse (1): Firing Time (cycles)	1st Pulse (1): Time Between Pulses	2nd Pulse: Heat Setting	2nd Pulse: Firing Time (cycles)	Hold Time (cycles)	Density (by Displacement) (5.47 g/cc = 100%) g/cc	Density (by Displacement) (5.47 g/cc = 100%) %	Rockwell Hardness (R_A)	Behavior and Appearance	Transverse Rupture Specimens: Modulus of Rupture (psi) (3)	Transverse Rupture Specimens: Density (by Displacement) g/cc	Transverse Rupture Specimens: Density (by Displacement) %	Transverse Rupture Specimens: Rockwell Hardness (R_A)
									secs.											
567	.450	5.0	3	8000		1	6.0	13	3.5	6.0	16	9				Partially sintered				
570							6.0	16		6.0	15	9				Sintered. Broken in center				
568							6.0	16		6.0	14	9				" Split specimen				
569							6.0	16		6.0	16	28				Minor expulsion. Crack in liner				
569							6.0	16		5.7	15	9				Sintered. Cracked on ejection				
571							6.2	16		5.7	15	9				" Split in center				
572							6.4	16		5.7	15	9				Extrusion. Cracked liner				
									cycles											
773	.720	8.4	3	7900	2900	8	4.0	16	2	5.0	10	36				Sintered	74,000	5.37	98.2	88-89
									secs.											
574	.450	5.0	3	6600		1	6.4	16	3.5	5.6	15	9				Not sintered				
575							6.8	16		5.6	15	9				" " Broken in center				
576							7.2	16		5.6	15	9				" "				
579							7.4	16		5.0	15	9	5.41	98.9	75-85	Sintered				
577-78	.410	4.76	3	6600		1	7.4	16	3.5	5.5	15	9				Expulsion				
580							7.4	16		5.2	15	9			83-87	Sintered				
582							7.5	16		5.2	15	9				Extrusion				
581							7.6	16		5.2	15	9				"				
656	.720	8.4	3	6350	3600	8	3.5	16	3.5	7.0	15	9				Sintered				
655					5000		3.5	16		8.0	15	9				Expulsion				
708	.720	8.4	3	6200	3400	8	6.6	15	3.7	3.5	16	9	5.42	99.1	85-83	Sintered				
709					3600		6.6	15		3.5	16	9	5.34	97.6		" . Slight extrusion				
710					3200		6.6	15		3.5	16	9	5.31	97.1		" " "				
711					3400		6.6	15		3.5	16	9	5.30	96.9	82-81	" " " . Badly cracked liner				
712					5400		6.6	15		3.5	16	9	5.15	94.1	85-85	Sintered	55,700	5.40	98.7	80-86
713					4000		6.6	15		3.5	16	9				Extrusion. Broken during extraction from die				
714					3800		6.6	15		3.5	16	9				" " " "				
715					3800		6.6	15		3.5	16	9	5.44	99.5	81-86	Sintered	67,000	5.42	99.1	84-86
716					3400		6.6	15		3.5	16	9	5.36	98.0	82-84	Extrusion. Due to loose fit in liner				
717					3400		6.6	15		3.5	16	9	5.43	99.3	83-87	Sintered				
718					3400		6.6	15		3.5	16	9				Slight extrusion. Broken on ejection				
722					3600		6.6	15		3.5	16	9	5.44	99.5	81-83	Sintered	63,200	5.40	98.7	86-87
724					3800		6.6	15		3.5	16	9	5.45	99.6	87-85	"	60,400	5.39	98.5	82-85
725					3700		6.6	15		3.5	16	9	5.36	98.0	83-80	"	49,200	5.40	98.7	85-88
726					3600		6.6	15		3.5	16	9	5.35	97.8	81-81	"				
707					3600		6.7	15		3.5	16	9				" . Some extrusion				
																Partial extrusion. Broken on ejection				
706					3800		6.8	15		3.5	16	9				Partial extrusion				
703					3500		6.9	15		3.5	16	9	5.29	96.7	81	Sintered				
704					3900		6.9	15		3.5	16	9	5.06	92.5	80-81	Expulsion of material				
705					4100		6.9	15		3.5	16	9	5.31	97.1		Partially sintered				
719					5000		6.9	15		3.5	16	9			80-82	Sintered. Ends only partially sintered	38,600	5.33	97.4	86-87
702					4000		6.9	17		3.5	16	9	5.30	96.9		Expulsion				
700					3800		6.9	20		3.5	16	9	5.43	99.3	80-84	" . Cracked specimen		5.36	98.0	(Specimen cracked)
701					3440		6.9	15		3.5	17	9				Partial extrusion. Porous specimen				
720					5000		7.2	15		3.5	16	9	5.44	99.5	85-86	Sintered. Liner broken	62,000	5.47	100	89-85
721					5200		7.2	15		3.5	16	9	5.45	99.6	84-85	"	59,000	5.23	95.6	84-86
723					5600		7.2	15		3.5	16	9	5.35	97.8		Partial extrusion				
									cycles											
675	.720	8.4	3	6200	4400	8	2.0	19	1	5.0	6	9				Not sintered				
676					4400		3.5	19		5.0	6	9				" "				
									secs.											
699	.720	8.4	3	6200	3700	8	3.5	15	3.7	6.9	16	9				Violent expulsion with arcing				
657	.720	8.4	3	6200	4400	8	3.5	16	3.5	6.8	15	9				Not sintered				
659					5000		3.5	16		6.8	15	9				" "				
658					4200		3.5	16		6.8	15	9				Sintered	87,000-80,000	5.38		87-88
659a					4200		3.5	16		6.8	15	9				"				
660					4000		3.5	16		6.9	15	9				"	143,000-173,000	5.39		87-89
									cycles											
677	.720	8.4	3	6200	3200	8	5.0	19	1	5.0	6	9				Not sintered. Only slightly compacted				
678					4600		5.0	19		6.0	6	9				" " " " "				
679					3800		5.0	19		6.5	6	9				Partially sintered				
682					3200		5.0	19		6.5	6	9	5.46	99.8	86-85	" " . Folds lengthwise in compact	(66,800 (83,100	5.39	98.5	89-89
680					4000		5.0	19		7.0	6	9				Partially sintered				
683					3600		5.0	19		7.0	6	9				" . Broken on hammer blow				
695					2800		5.0	19		7.0	6	5	5.41	98.9	83-86	Sintered. Porous. Ends soft				
698					3000		5.0	19		7.0	6	8				Not sintered				
696					3000		5.0	19		7.0	6	10	5.43	99.3	85-85	Sintered. Ends soft	56,700	5.37	98.2	83-84
697					3000		5.0	19		7.0	6	11				" . Some extrusion				
684					3400		5.0	19		7.4	6	9	5.46	99.8	85-85	" . Folds lengthwise in compact	(84,500 (79,900	5.40	98.7	86-88
685					3600		5.0	19		7.8	6	9	5.43	99.3	78-85	" " " " " "	(113,000 (38,500	5.35	97.8	85-87
693					4400		5.0	19		7.6	6	9				Partially sintered				
681					4600		5.0	19		8.0	6	9				" "				
686					3600		5.0	19		8.2	6	9				" " . Broken at light hammer blow				
689					3600		5.0	19		8.2	6	5	5.43	99.3	73-80	Sintered	(87,700 (77,900 (116,000	5.37	98.2	85-87
692					3600		5.0	19		8.2	6	9				Expulsion				
687					3600		5.0	19		8.6	6	9				Sintered. Broken at joint				
690					4000		5.0	19		8.6	6	28				Minor expulsion through crack in liner				
691					3600		5.0	19		8.6	6	9				Expulsion				
688					3800		5.0	19		9.0	6	9	5.43	99.3	70-85	Blistered	121,000	5.40	98.7	84-86
									secs.											
669	.720	8.4	3	6200	3600	8	6.5	16	3.5	3.5	15	9	5.43	99.3	86	Center sintered. Ends only partially sintered. Too cold				
670					3600		6.7	16		3.5	15	9	5.45	99.6	85-87	Center sintered. Ends upset	(44,500 (68,500 (55,700	5.41	98.9	68-83
672					4200		6.7	16		3.5	15	9	5.46	99.8	83-85	Sintered	(65,100 (71,600	5.42	99.1	87
673					5000		6.7	16		3.5	15	9	5.47	100.0	81-65	" . Slight extrusion at ends	62,300	5.37	98.2	88-88
674					5000		6.7	16		3.5	15	9	5.42	99.1	70	" " " " " "				
671					4200		6.9	16		3.5	15	9				Expulsion of material				
594	.640	7.5	2	4100		1	9.4	16	3.5	3.8	15	9	5.42	99.1	86-87	Sintered				
595							9.4	16		3.8	15	9	5.48	99.5	83-87	"				
596							9.4	16		3.8	15	9	5.42	99.1	70-86	"				
597							9.4	16		3.8	15	9	5.42	99.1	85-88	"				
611					4500		9.4	16		3.8	15	9				Partially sintered				
612					4500		9.4	16		3.8	15	9				"				
613					2800		9.4	16		3.8	15	9				Sintered. Blistered				
586	.410	5.0	3	4100		1	9.6	16	3.5	3.0	15	9	5.38	98.4	87-55	Sintered. Ends soft				
592							9.6	16		3.8	15	9	5.43	99.3	80-87	"				
587							9.6	16		4.0	15	9				" . Broken by hammer blow				
588							9.6	16		4.0	15	9	5.45	99.6	83-87	Sintered. Bulge at center				
583							9.6	16		5.0	15	9				" . Some bulging				
584							9.6	16		5.0	15	9	5.45	99.6	85-89	"				
635	.720	8.4	3	4100	5400	8	3.0	16	3.5	8.4	15	9				Sintered. Some bulging				
640					4900		3.0	16		8.6	15	9				" . Partially bulged	(53,300 (74,900	5.28		87-87
633					4600		4.0	16		8.0	15	9				Center overheated				
632					5000		8.0	16		5.6	15	9				" "				
626					4500		8.6	16		5.1	15	9				Sintered	(60,900 (49,800 (78,500	5.40		88-88
620					6200		8.9	16		2.8	15	9				" . Some bulging of compact	(72,500 (29,600 (61,400	5.35		87-89
623					5200		8.9	16		5.0	15	9				Sintered. Rough surface	134,200	5.30		87-89
625					5000		8.9	16		5.1	15	9				" " "				
624					5000		8.9	16		6.0	15	9				Expulsion				

NOTES:

(1) Samples are arranged in order of increasing heat and/or time of 1st. pulse within each group.

(2) Carbowax No. 400 (Union Carbide and Carbon Corporation) used as powder lubricant instead of paraffin in Samples No. 570 through 579.

(3) Duplicate transverse rupture tests were run where sample was of sufficient length.

TABLE IV (Continued)

TEST RESULTS OF FLASH SINTERING OF 80% TITANIUM CARBIDE - 20% "F" CARBONYL NICKEL COMPACTS (1 HOUR PRESINTER)

Specimen Number	Nominal Length of Presintered Compacts Before Flash Sintering (in.)	Nominal Weight (gms.)	Number of Compacts Used	Ram Load (lbs.)	Resistance of Assembly (At Ram Load) (micro ohms)	Voltage Tap	Single Pulse Heat Setting (1)	Single Pulse Firing Time (cycles)	Hold Time (cycles	Density (by Displacement) (5.47 g/cc = 100%) g/cc	%	Behavior and Appearance
891	.720	8.4	3	12000	3200	8	1.5	38	53			Not sintered
892					3400		2.4	38	53			Not sintered
893					3400		3.0	38	53			Not sintered
894					3400		4.0	38	53			Center sintered. Ends soft
897					3600		4.2	38	53			Center blistered
895					3200		4.5	38	53			Center extruded
876					2400		10.0	3	56			Not sintered
877					1900		10.0	6	56			Not sintered
880					2500		10.0	7	56	5.40	98.7	Sintered (4)
881					2500		10.0	7	56	5.44	99.5	Sintered (4)
878					2600		10.0	10	56	5.15	94.2	Expulsion
879					2500		10.0	21	56			Violent expulsion
907	.720	8.4	1	6200	3000	1	2.0	29	53			Not sintered
908					3000		3.0	29	53			Not sintered
909					3000		4.0	29	53			Sintered. Split in center
912					3000		4.2	29	53			Sintered. Split in center
910					3000		4.4	29	53			Sintered
911					3100		4.4	29	53			Sintered
904					3000		6.0	15	53			Sintered. Split in center
924					3100		6.0	15	53			Sintered. Split in center
926					3100		6.3	15	53			Sintered. Split in center
927					3000		6.3	15	53			Sintered. Partial extrusion
928					3200		6.3	15	53			Sintered. Split in center
929					3100		6.6	15	53			Sintered
925					3200		6.6	15	53			Extrusion
903							7.0	15	53			Expulsion
930					3100		7.0	15	53			Extrusion and expulsion
905					3100		8.6	6	53			Soft ends. No wafers
906					3000		8.6	6	53			Sintered. Split in center
913					3000		9.0	6	53			Sintered center. Soft ends
915					3000		9.4	6	53			Sintered. Center split
916					3000		9.6	6	53			Sintered. Center split
917					3000		9.8	6	53			Sintered
918					3000		10.0	6	53			Sintered
919	.720	4.2	2	6200	3000	2	9.3	6	53			Sintered. Blistered
921					3000		9.3	6	53			Extrusion
922					3000		9.3	6	53			Extrusion
923					3200		9.3	6	53			Sintered
920					3000		9.6	6	53			Sintered. Blistered
898	.720	8.4	3	3300	5200	8	4.5	38	53			Not sintered
900					4800		5.5	38	53			Center sintered. Ends soft
899					5400		6.0	38	53			Sintered
901					6800		6.0	38	53			Center sintered. Ends soft
902					6400		6.5	38	53			Sintered. Friable
845					4900		10.0	4	33			Not sintered
874					4800		10.0	17	56	5.37	98.2	Sintered. Friable
872					5000		10.0	18	56	5.25	96.0	Sintered. Friable
847					5400		10.0	29	33			Violent expulsion
802	.720	8.4	1	6200		1	7.0	7	64			"Sicon" coated (2). Eroded brass liner at wafers
803							7.0	16	64			"Sicon" coated. Eroded brass liner at wafers
804							7.0	16	64			Al_2O_3 / EC969 coated (3). Compact soft but could be ejected
805							7.4	16	64			Harder than Compact 804. Otherwise the same
806							7.4	16	64			Same coating. Soft on outside and ends. Good in center
807							7.8	16	64			Same coating. Extrusion

NOTES:

(1) Samples are arranged in order of increasing heat and/or time within each group.

(2) "Sicon" Plastic - Midland Industrial Finishes Company.

(3) EC-369 Plastic - Minnesota Mining & Manufacturing Company.

(4) Transverse Rupture Specimens

Specimen No. 880	Specimen No. 881

TABLE IV (Continued)

TEST RESULTS OF FLASH SINTERING OF 80% TITANIUM CARBIDE - 20% "F" CARBONYL NICKEL COMPACTS (1 HOUR PRESINTER)

Specimen Number (3)	Nominal Length of Presintered Compacts Before Flash Sintering (in.)	Nominal Weight (gms.)	Number of Compacts Used	Ram Load (lbs.)	Resistance of Assembly (At Ram Load) (micro ohms)	Voltage Tap	1st Pulse (1) Heat Setting	1st Pulse (1) Firing Time (cycles)	Time Between Pulses (cycles)	2nd Pulse Heat Setting	2nd Pulse Firing Time (cycles)	Hold Time (cycles)	Density (by Displacement) (5.47 g/cc = 100%) g/cc	Density (by Displacement) (5.47 g/cc = 100%) %	Rockwell Hardness (R_A)	Behavior and Appearance	Transverse Rupture Specimens: Modulus of Rupture (psi) (2)	Transverse Rupture Specimens: Density (by Displacement) g/cc	Transverse Rupture Specimens: Density (by Displacement) %	Transverse Rupture Specimens: Rockwell Hardness (R_A)
883	.720	8.4	3	12000	2400	8	10.0	5	2	2.0	5	56				Not sintered				
888					2300		10.0	7		1.0	45	56				Sintered. Extrusion through liner cracks	56,700			84
889					2200		10.0	7		1.0	85	56				Sintered. Friable				
886					2600		10.0	7		2.0	85	56	5.44	99.5		Expulsion				
882					2200		10.0	7		2.0	85	56	5.34	97.6		Extrusion	58,000			84
887							10.0	7		4.0	45	56	5.40	98.7		Expulsion				
786	.720	8.4	3	7900	3440	8	4.0	10	2	5.0	15	64	5.32	97.3		Not sintered				
787					3600		4.0	10		5.6	15	64	5.39	98.5	84-87	Ends not sintered	65,000	5.35	97.8	90-91
771					3000		4.0	16		5.0	10	64	5.40	98.7	89-85	Sintered	53,600	5.37	98.2	89-90
789					3000		4.2	10		5.6	15	64	5.38	98.4	83-86	Sintered. Center rough	84,000	5.34	97.6	90-86
792					3600		4.2	10		5.6	15	64	5.29	96.7	84-89	Sintered	55,700	5.36	98.0	88-90
793					3400		4.2	10		5.6	15	64	5.42	99.1	84-90	Ends not sintered. Center rough	81,400	5.37	98.2	88-90
794					3000		4.2	10		5.6	15	64				Overheated. Cracked on approval				
795					3200		4.2	10		5.7	15	64	5.43	99.3	80-87	Sintered	77,600	5.36	98.0	88-90
796					3200		4.2	10		5.7	15	64				Sintered. Friable				
797					3500		4.2	10		5.7	15	64				Sintered. Friable				
798					3200		4.2	10		5.7	15	64				Sintered. Friable				
799					3200		4.2	10		5.7	15	64	5.42	99.1	83-86	Sintered	99,200	5.36	98.0	90
800					3100		4.2	10		5.7	15	64	5.39	98.5	84-88	Sintered	90,500	5.36	98.0	88-90
801					3500		4.2	10		5.7	15	64	5.43	99.3	84-87	Sintered. Ends not sintered	63,300	5.37	98.2	88-90
791					4600		4.2	10		6.4	15	64	5.44	99.5	79-83	Sintered. Center rough. Cold ends	75,200	5.38	98.4	88-91
788					3400		4.4	10		5.6	15	64				Slight extrusion				
767					2900		6.0	16		2.6	15	64				Cracked liner. Sintered				
768					2900		6.0	16		2.6	10	64				Broken on removal from holder				
769					3000		6.0	16		2.6	10	64				Broken on removal from holder				
770					3000		6.0	16		2.6	10	64				Broken on removal from holder				
766					2700		6.0	16		3.0	10	64				Extrusion				
765					2700		6.0	16		3.2	10	64				Slight extrusion. Broken on hammer blow				
764					3000		6.0	16		3.6	10	64				Slight extrusion. Broken on hammer blow				
860	.720	8.4	3	6200	4800	8	3.5	15	secs. 3.1	4.0	18	23				Not sintered				
850					3500		3.5	15		4.5	18	23				Not sintered				
851					4400		3.5	15		5.2	18	23				Not sintered				
852					3400		3.5	15		5.2	18	23	5.40	98.7		Sintered	86,000	5.36	98.0	88
853					4000		3.5	15		5.6	18	23	5.41	98.9		Sintered	(63,300 (57,800 (56,500	5.39	98.5	88
854					3500		3.5	15		5.6	18	23	5.40	98.7		Sintered	58,900 48,300	5.39	98.5	86.5
856					3800		3.5	15		5.6	18	23	5.43	99.3		Sintered				
857					3400		3.5	15		5.6	18	23	5.46	99.9		Sintered. End broken on hammer blow	90,100 80,200	5.38	98.4	89
861					4000		3.5	15		5.6	18	23	5.42	99.1		Not quite sintered. Broken on hammer blow				
862					3900		3.5	15		5.6	18	23	5.38	98.4		Sintered. Broken on hammer blow				
863					3400		3.5	15		5.7	18	56	5.38	98.4		Sintered. Broken on hammer blow				
870					3800		3.5	15		5.7	18	56	5.42	99.1		Sintered. End chipped on hammer blow	90,000 102,200 92,500	5.37	98.2	88.5
859					3500		3.5	15		5.8	18	23	5.44	99.5		Sintered. Slight flow hole on end. Broken on hammer blow				
868					3800		3.5	15		5.8	18	23	5.38	98.4		Sintered. Broken on hammer blow				
855					4200		3.5	15		6.0	18	23	5.40	98.7		Extruded through flow hole	59,900 69,500	5.40	98.7	88
858					4000		3.5	15		6.0	18	23	5.42	99.1		Sintered. Broken on hammer blow				
865					3700		3.5	15		6.0	18	23	5.38	98.4		Expulsion	118,000 99,500	5.28	96.5	87.5
864					3600		3.5	15		6.0	18	23	5.43	99.3		Sintered. Broken on hammer blow				
866					3800		3.5	15		6.0	18	23				Sintered				
867					3800		3.5	15		6.0	18	23	5.40	98.7		Sintered. Brokwn on hammer blow				
865					3600		3.5	15		6.0	18	56	5.37	98.2		Sintered	110,500 101,900	5.37	98.2	87.5
849					3700		3.5	15		6.9	18	23				Violent expulsion				
772	.720	8.4	3	6200	4400	8	4.0	16	cycles 2	5.0	10	64				Not sintered				
773					2900		4.0	16		5.0	10	64	5.39	98.5	85-88	Sintered	74,000	5.36	98.0	88-89
752					3600		5.0	16		6.8	7	64				Sintered. Broke on removal from holder				
753					4400		5.0	16		6.8	7	64			82-89	Ends not sintered	77,000	5.36	98.0	88-89
758					4000		6.0	16		4.0	10	64				Sintered. Liner cracked				
759					3400		6.0	16		4.0	10	64	5.39	98.5	84-85	Sintered	50,600	5.35	97.8	86-90
760					3400		6.0	16		4.0	10	64	5.34	97.6	82-83	Extrusion through cracked liner	112,000	5.34	97.7	87-90
762					3800		6.0	16		4.0	10	64	5.42	99.1	84-86	Sintered	64,000 63,500	5.36	98.0	89-90
763					3800		6.0	16		4.0	10	64				Broken on removal from holder				
761					4600		6.0	16		4.2	10	64	5.38	98.4	78-83	Sintered. Some expulsion near end of heat				
754					3600		6.0	16		5.8	7	64				Ends sintered. Center rough				
755					5200		6.0	16		5.8	7	64				Some expulsion. Cracked liner				
756					4800		6.0	16		5.8	7	64				Ends not sintered				
757					3600		6.0	16		5.8	7	64				Sintered. Mild expulsion				
750	.720	8.4	3	6200	4200	8	7.2	15	secs 3.8	3.5	16	10				Cracked. Partly sintered				
747					4600		7.6	15		3.5	16	10				Sintered				
748					5000		7.7	15		3.5	16	10				Cracked. Vikent expulsion				
749					5000		7.7	15		3.5	16	10	5.29	96.7		Violent expulsion on first pulse				
790	.720	8.4	3	3000	3400	8	4.2	10	cycles 2	5.6	15	64	5.42	99.1	83-84	Sintered. Center rough	63,400	5.37	98.2	89-90
				(4000 First Pulse																
				(8000 Second Pulse																
808	.720	8.4	3	(	4400	8	4.4	15	2	6.0	10	64				Not sintered				
828					3600		4.8	15		6.4	10	64	5.43	99.3		Sintered				
831					3800		4.8	15		6.5	10	64				Ends fairly sintered. Broken on hammer blow				
824					3600		4.8	15		6.5	10	64	5.34	97.6		Extrusion				
818					3800		4.8	15		6.6	10	64	5.40	98.7		Sintered				
819					3800		4.8	15		6.6	10	64	5.35	97.8		Sintered				
820					3700		4.8	15		6.6	10	64	5.41	98.9		Extrusion				
821					4000		4.8	15		6.6	10	64	5.42	99.1		Ends not fully sintered				
825					4000		4.8	15		6.6	10	64				Sintered. Broken on hammer blow				
826					3600		4.8	15		6.6	10	64	5.35	97.8		Extrusion. Broken on machining				
827					4000		4.8	15		6.6	10	64	5.42	99.1		Sintered	48,500	5.33	97.5	87
829					4000		4.8	15		6.6	10	64	5.39	98.5		Sintered				
822					4200		4.8	15		6.7	10	64	5.47	100.0		Ends not fully sintered. Broken on hammer blow				
823					4000		4.8	15		6.7	10	64	5.32	97.3		Extrusion. Brokwn on machining				
830					4600		4.8	15		6.8	10	64				Not fully sintered. Broken on hammer blow				
832					4400		4.8	15		6.9	10	64				Sintered. Broken at glassy extrusion				
833					4500		4.8	15		6.9	10	64	5.41	98.9		Sintered	44,500	5.41	98.9	87
835					4000		4.8	15		7.0	10	64	5.40	98.7		Sintered	49,800	5.36	98.0	87.5
837					3800		4.8	15		7.0	10	64	5.34	97.6		Sintered. Extrusion in broken liner	44,500	5.37	98.2	87.5
839					3900		4.8	15		7.1	10	64	5.43	99.3		Sintered. Ends not fully sintered	32,400	5.31	97.2	87.5
834					4500		4.8	15		7.2	10	64				Sintered. Cracked in center. Soft ends				
838					4200		4.8	15		7.2	10	64	5.40	98.7		Sintered	62,000	5.37	98.2	87.5
840					4200		4.8	15		7.3	10	64	5.39	98.5		Sintered	41,800	5.37	98.2	87
841					4000		4.8	15		7.3	10	64	5.40	98.7		Sintered				
842					4000		4.8	15		7.3	10	64	5.43	99.3		Sintered	47,200	5.41	98.9	87
843					4200		4.8	15		7.5	10	64	5.34	97.6		Sintered	51,300	5.40	98.7	85.5
844					4000		4.8	15		7.5	10	64	5.39	98.5		Sintered	63,400	5.40	98.7	87
836					4800		4.8	15		7.8	10	64				Ends soft. Broke with difficulty				
810					4000		5.0	15		6.0	10	64				Ends sintered. Center blistered. Glassy extrusion. New liner stuck				
815					4400		5.0	15		6.6	10	64				Not sintered				
817					4000		5.0	15		6.6	10	64	5.34	97.6		Very mild extrusion	62,000	5.36	98.0	87.5
816					4600		5.0	15		6.6	12	64				Extruded at end of last pulse. Specimen cracked				
811					4000		5.0	15		7.0	10	64				Extrusion				
812					4200		5.0	15		7.0	10	64				Cracked specimen - extrusion				
813					4800		5.0	15		7.0	10	64				Expulsion. Glassy extrusion at break				
814					6000		5.0	15		7.0	10	64				Not sintered				
809					4300		5.4	15		6.0	10	64				Cold ends - hot center. Extruded into liner crack				

NOTES:

(1) Samples are arranged in order of increasing heat and/or time of 1st. pulse within each group.

(2) Duplicate transverse rupture tests were run where sample was of sufficient length.

(3) Paraffin added to carbon tetrachloride carrier before milling in all cases except Specimen Nos. 856 through 865 for which paraffin was added after milling and before drying.